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LEEDS & WEST RIDING

MEDICO-CHIRURGICAL SOCIETY

# PUBLIC HEALTH LABORATORY WORK

BY

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D.P.H., F.C.S.

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THE PARISH OF STOKE NEWINGTON; FELLOW OF THE  
SANITARY INSTITUTE ETC.

SECOND EDITION

WITH ILLUSTRATIONS

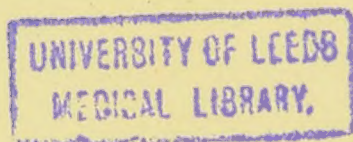
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1896



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## PREFACE.

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THE issue of a second edition of this work has furnished the opportunity to submit it to a thorough revision; some processes given in the former edition have been replaced by others, and a considerable amount of new material has been added without increasing the size of the book.

It is hoped that these pages will continue to prove of value to those interested in Public Health and to those seeking Public Health Degrees, and that they may be read along with the many excellent works upon Hygiene and Public Health which are now in circulation, but which of necessity do not deal fully with the subject of Hygienic Analysis.

To treat the subject exhaustively would necessitate a very bulky volume, but an effort has been made to convey to the reader, in a concise and practical form, the knowledge necessary to enable him to best perform those analyses which may be

fairly considered to be included within the domains of practical hygiene.

The writer acknowledges his indebtedness to the useful bacteriological notes by Dr. CHRISTOPHER CHILDS, for he recognises how much they enhance the value of the work ; he also desires to express his thanks to Professor CORFIELD, and Drs. LOUIS PARKES, BRAGA, SLATER and BULLOCK for valuable suggestions and advice, and to Messrs. TOWNSON and MERCER and others for the loan of many wood blocks.

H. R. K.

HYGIENIC LABORATORY,

UNIVERSITY COLLEGE, W.C.

*March, 1896.*



# CONTENTS.

---

## PART I.

### THE HYGIENIC ANALYSIS OF WATER.

#### CHAPTER I.

	PAGE
The fitting up of a laboratory for water analysis . . .	1
Table of atomic weights . . . . .	15
Weights and measures upon the metrical system . . .	16

#### CHAPTER II.

A water report . . . . .	18
--------------------------	----

#### CHAPTER III.

The collection and labelling of samples . . . . .	21
---	----

#### CHAPTER IV.

The analysis . . . . .	27
The physical characters . . . . .	28

#### CHAPTER V.

Chlorine . . . . .	34
--------------------	----

#### CHAPTER VI.

Hardness . . . . .	41
Quantitative test for temporary and permanent hardness . .	44
Hardness due to magnesium salts . . . . .	49

#### CHAPTER VII.

The poisonous metals . . . . .	51
--------------------------------	----

## CHAPTER VIII.

PAGE.

The non-poisonous metals . . . . .	65
Sulphates . . . . .	68
Phosphates . . . . .	69

## CHAPTER IX.

The solid residue . . . . .	72
Suspended matter . . . . .	78

## CHAPTER X.

Organic matter in water . . . . .	80
-----------------------------------	----

## CHAPTER XI.

Wanklyn's process . . . . .	85
-----------------------------	----

## CHAPTER XII.

The oxidisable organic matter . . . . .	99
---	----

## CHAPTER XIII.

Frankland's process . . . . .	106
-------------------------------	-----

## CHAPTER XIV.

Oxidised nitrogen (nitrates and nitrites) . . . . .	110
---	-----

## CHAPTER XV.

Examination of the sediment . . . . .	124
Chemical examination . . . . .	124
Microscopic examination . . . . .	125

## CHAPTER XVI.

Gases in water . . . . .	133
Analysis of mineral waters . . . . .	144
The opinion upon a water analysis . . . . .	147



## CONTENTS.

xi

### CHAPTER XVII.

	PAGE
Water samples . . . . .	151
A very pure water . . . . .	151
A foul and dangerous water . . . . .	152
A suspicious water . . . . .	153
Rain water . . . . .	154
A typical peaty "upland surface" water . . . . .	155
Spring water from the chalk . . . . .	156
A typical rain water . . . . .	156
A deep well water from the chalk . . . . .	158
Sea water . . . . .	158
Scheme for performing water analysis . . . . .	159
An analytical table . . . . .	161
Sewage effluents . . . . .	164
Alkalimetry and acidimetry . . . . .	165

---

## PART II.

### SOIL EXAMINATION.

The analysis and classification of soils . . . . .	168
Manures . . . . .	179

---

## PART III.

### AIR ANALYSIS.

#### CHAPTER I.

The normal and abnormal constituents of air . . . . .	180
Town and country air . . . . .	181
Eudiometry . . . . .	185

#### CHAPTER II.

Collecting samples . . . . .	191
Examination by the senses . . . . .	194
Reaction . . . . .	195

## CHAPTER III.

	PAGE
Carbonic acid . . . . .	196

## CHAPTER IV.

The organic matter in air . . . . .	208
-------------------------------------	-----

## CHAPTER V.

Ammonia . . . . .	212
Marsh gas . . . . .	213
Carbonic oxide . . . . .	213
Sulphurous and sulphuric anhydride, sulphuretted hydrogen, ammonium sulphide . . . . .	220
Hydrochloric acid, nitric and nitrous acids, carbon bisulphide	221
Table of injurious amounts of gaseous impurities . . . . .	222

## CHAPTER VI.

Ozone . . . . .	223
Aqueous vapour . . . . .	226
Peroxide of hydrogen . . . . .	229

## CHAPTER VII.

Suspended matter in the air . . . . .	230
The methods of collection . . . . .	231
The nature of the suspended matter . . . . .	234

## CHAPTER VIII.

The characters of the air collected from various sources . . . . .	237
Marsh air . . . . .	237
Sewer air . . . . .	237
Air over burial grounds . . . . .	238
Air of mines . . . . .	239
Air during fogs . . . . .	239
Ground air . . . . .	239



# CONTENTS.

xiii

## CHAPTER IX.

	PAGE
Scheme for the detection of gases when present in large quantities . . . . .	242

## PART IV.

### COAL GAS ANALYSIS.

The composition and analysis of coal gas . . . . .	246
The gaseous compounds of sulphur . . . . .	248
Ammonia . . . . .	253
Carbonic acid and oxide . . . . .	254
Tar . . . . .	254

## PART V.

### FOOD EXAMINATION.

#### CHAPTER I.

Composition of cow's milk . . . . .	256
The milk of diseased cows . . . . .	260

#### CHAPTER II.

The analysis of milk . . . . .	262
The physical characters . . . . .	262
The cream . . . . .	266
The specific gravity . . . . .	267
The total solids and the ash . . . . .	269
Solids-fat and non-fat . . . . .	270
Microscopic examination . . . . .	277

#### CHAPTER III.

Adulteration of milk . . . . .	279
Skimmed milk . . . . .	285

## CHAPTER IV.

	PAGE
Butter . . . . .	286
The analysis . . . . .	287
Butter substitutes and their identification . . . . .	288
Cheese . . . . .	302
Parasitic growths of . . . . .	303
Tyrotrocon . . . . .	305
Lard . . . . .	306

## CHAPTER V.

Corn . . . . .	307
Animal and vegetable parasites in . . . . .	307
Wheat-flour . . . . .	312
The analysis . . . . .	312
Adulteration . . . . .	314

## CHAPTER VI.

Bread . . . . .	316
Baking powders . . . . .	316
The analysis . . . . .	317
Adulteration . . . . .	318
Microscopic examination . . . . .	319
Alum in bread . . . . .	320

## CHAPTER VII.

The average composition of other cereals and the pulses . . . . .	325
Oatmeal . . . . .	325
Arrowroot . . . . .	325
Sago . . . . .	326
Tapioca . . . . .	326
Cornflour . . . . .	326
Lentil flour . . . . .	327
Pea meal . . . . .	327
Bean meal . . . . .	327
Rye meal . . . . .	328
Barley meal . . . . .	328
Potato flour . . . . .	328
Ground rice . . . . .	329
The microscopic characters of the different starch granules, &c. . . . .	329



LEEDS & WILSTEDING  
 MEDICAL POPULAR SOCIETY  
 CONTENTS.

CHAPTER VIII.

	PAGE
Meat . . . . .	339
The characters of good meat . . . . .	340
The characters of bad meat . . . . .	342
Frozen meat . . . . .	345
Opinion to be formed with regard to condemnation . . . . .	345
The important parasites of flesh . . . . .	350
Cysticerci . . . . .	350
Hydatids . . . . .	352
Trichina spiralis . . . . .	353
Actinomycosis . . . . .	356
Distoma hepaticum . . . . .	357
Life histories of the important animal parasites of man . . . . .	358
Fish . . . . .	361
Horseflesh . . . . .	362
Sausages, pork pies, meat pies, brawn, &c. . . . .	363
Eggs . . . . .	365

CHAPTER IX.

Alcoholic beverages . . . . .	366
Spirits . . . . .	367
Fusel oil . . . . .	367
Beer . . . . .	369
Wine . . . . .	371
Vinegar . . . . .	375
Mustard . . . . .	376
Pepper . . . . .	377
Sugar . . . . .	379

CHAPTER X.

Coffee . . . . .	382
Chicory . . . . .	382
Cocoa . . . . .	385
Chocolate . . . . .	387
Microscopic characters of coffee, chicory and cocoa . . . . .	387

## CHAPTER XI.

PAGE

Tea . . . . .	390
Infants' foods . . . . .	397

## CHAPTER XII.

Preserved and tinned provisions . . . . .	399
Antiseptics employed in food . . . . .	401
Colouring agents employed in food . . . . .	403

## CHAPTER XIII.

The detection of poisons in food . . . . .	410
Phosphorus . . . . .	411
Hydrocyanic acid . . . . .	412
Strychnine . . . . .	414
Atropine . . . . .	414
Arsenic . . . . .	415
The examination of wall-papers, &c., for arsenic . . . . .	416

## CHAPTER XIV.

Disinfectants . . . . .	419
-------------------------	-----

**PART VI.**

By CHRISTOPHER CHILDS, M.A., M.D., D.P.H.

Bacteriological examination of air, water, soil, food, &c. . . . .	425
--	-----

PLATE I.



*Volvox*



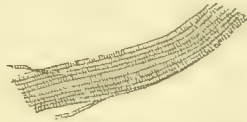
*Paramecium*



*Ova of Terebra Solium*



*Epithelial Cells*



*Voluntary Muscular Fibres*



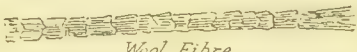
*Human Hair*



*Cotton Fibres*



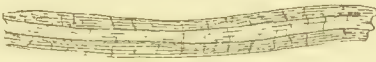
*Linen Fibres*



*Wool Fibre.*



*Silk Fibre.*



*Hemp Fibre*



*Particle of Feather*



*Pus Corpuscles*



*Pus Corpuscles  
(treated with Acetic Acid)*



*Human Blood  
Corpuscles*



*do (sideways)*



*do (crenated &  
forming a rouleau)*



*Crystals of Sodium Chloride  
(in air)*



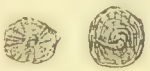
*Particles of Soot  
(in air)*



*Human Hair*



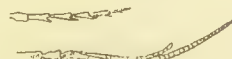
*Gammarus  
Pulex*



*Scales of Fish.*



*Amoeba*



*Antenna of Insects*





# LECTURES & READING

## IN THE ZOOLOGICAL SOCIETY



*Paramecium*



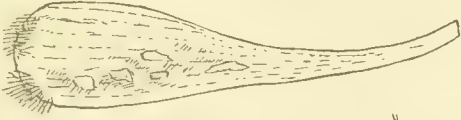
*Tricena*



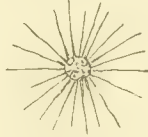
*Coleps*



*Gyrodinium*



*Stentor*



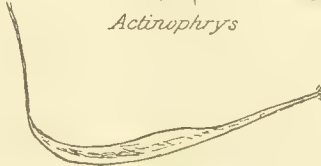
*Actinophrys*



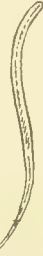
*Oxytricha*



*Amphileptus*



*Tracheocerca*



*Anguillula*



*Rotifer*



*Fresh Water Hydra*



*Cyclops*



*Scale of Insect*



*Daphnia*



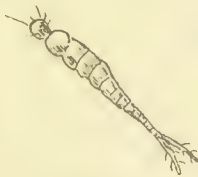
*A Water Mite*



*Part of leg of Cockroach*



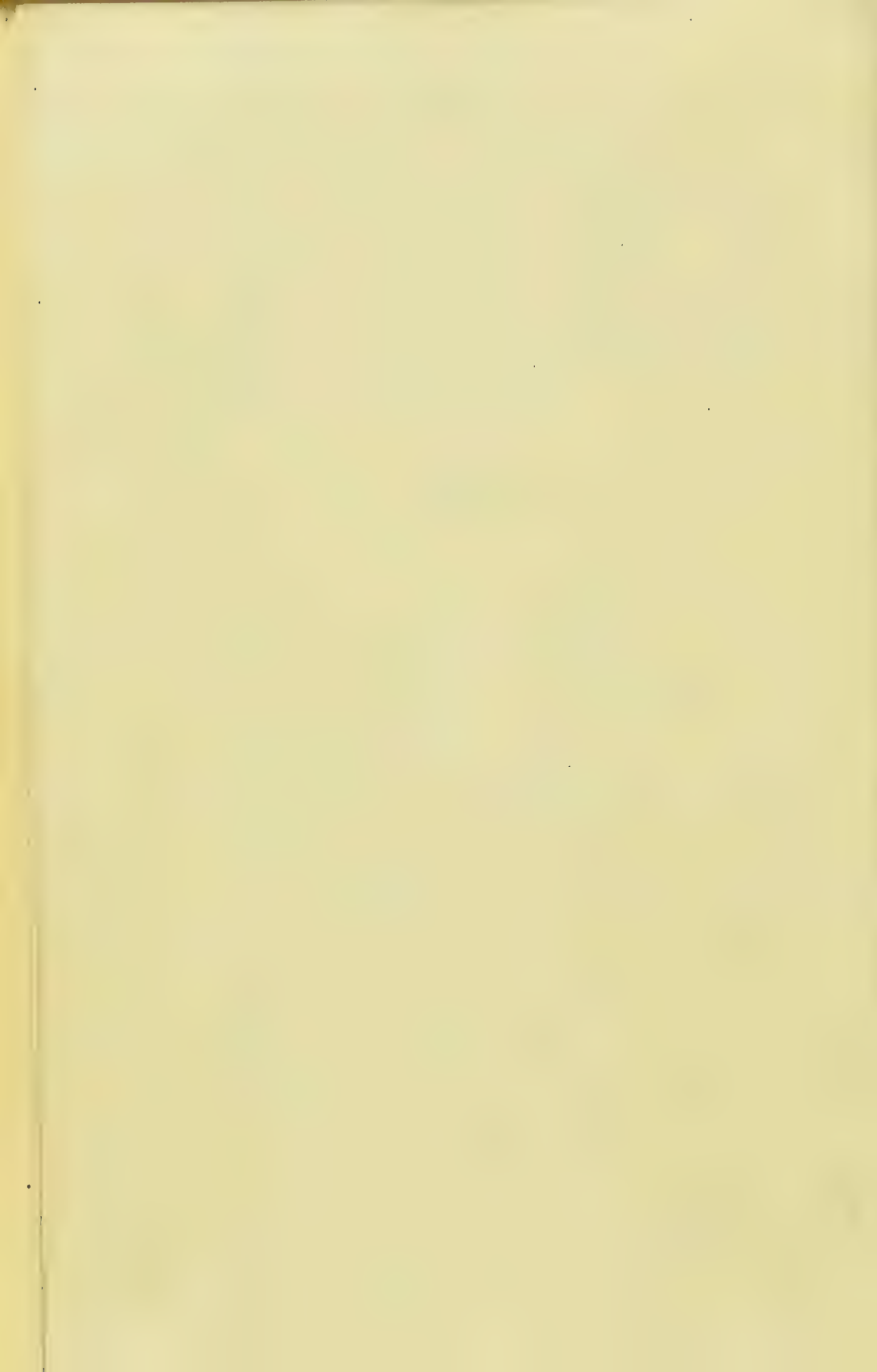
*A Water-bear*



*Lava of Insect*



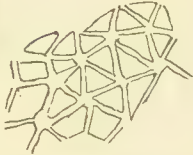
*Pupa of Insect*







*Cuticle of Grass with Mycelium*



*Form of Vegetable Tissue*



*Beggiatoa Alba*



*Pollen of Water Plantain*



*Spicules of Spongilla Fluviatilis*



*A Desmid*



*Vegetable Spore*



*Protococcus Fluviatilis*



*Pollen*



*Bacterium termia*



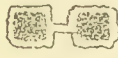
*Monas*



*Tabellaria flocculosa*



*Spores of Fungi*



*A Diatom*



*Surirella*



*Euglena Viridis*



*A Conterovoid cell.*



*A Desmid*



*Fungus*



*Oscillaria*



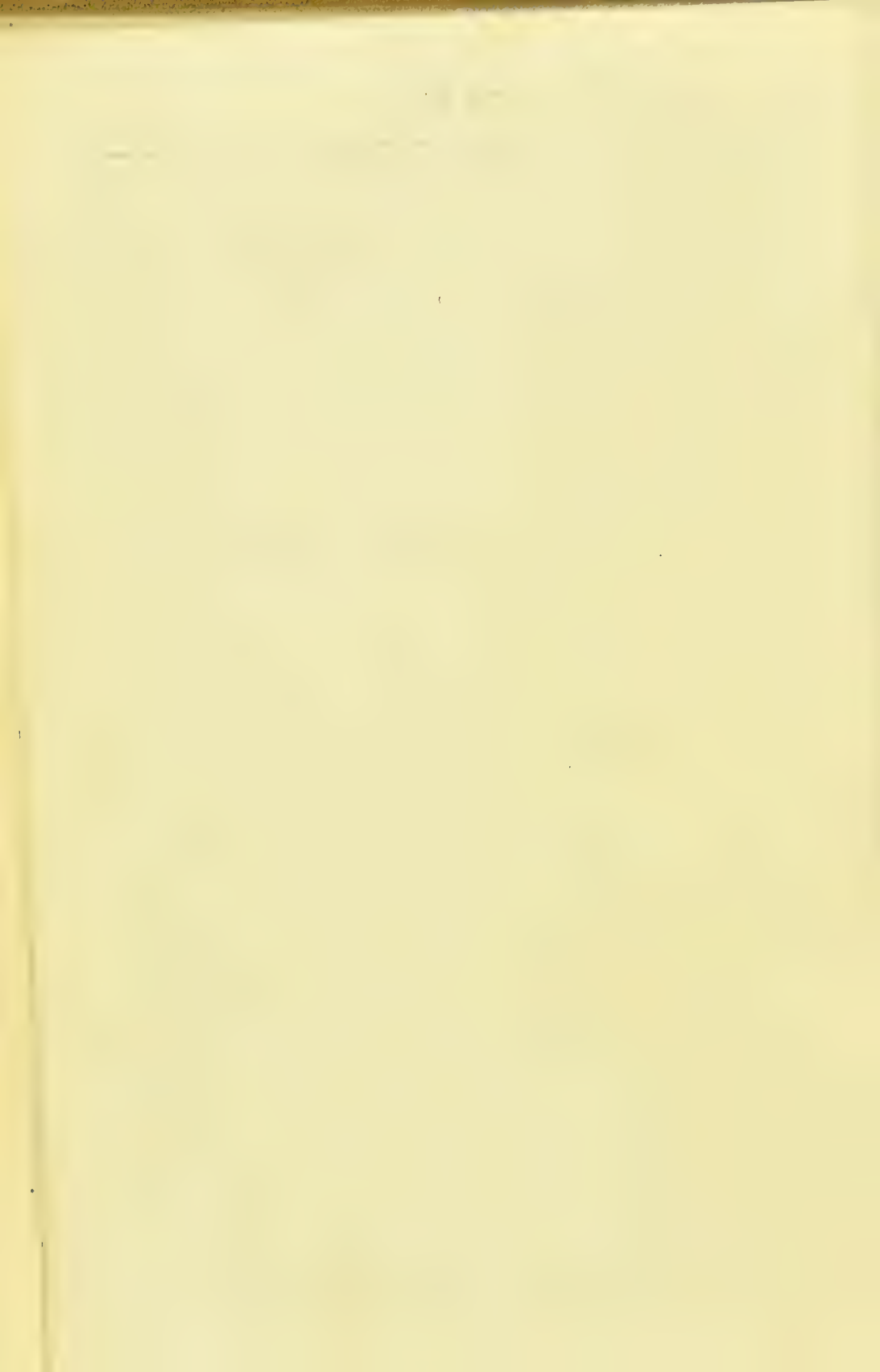
*Vegetable Tissue*



*Conferva*



*Gomphonema*



# PUBLIC HEALTH LABORATORY WORK.

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## PART I.

### THE HYGIENIC ANALYSIS OF WATER.

#### CHAPTER I.

##### THE LABORATORY.

THE object of a hygienic analysis of water is to learn whether the use of such for drinking purposes should be sanctioned or condemned, from a health standpoint.

If it is desired to fit up a laboratory to meet all the requirements of a hygienic analysis of water, it will be necessary to provide in the first place a bench, six feet long and three feet broad, standing to half the height of the operator, and placed in a good light; this must be fitted up at the back with shelves, at least capable of carrying those chemical reagents which are in most common use. By the side of the bench there should be a tap (furnishing a good supply of pure water), with a small water sink beneath it. There must be a supply of coal gas conducted to the bench so as to serve two or more separate Bunsen burners. The Bunsen burner (fig. 1) consists of a larger tube surrounding the base of a smaller gas-delivering one, the former being perforated for the admittance of air—so that at the top the gas escapes well mixed with air; this has the effect of increasing oxidation (and therefore heat) in the flame. Fletcher's burners

are an improvement upon the common types of Bunsen burners, where it is required to employ a very small flame.

**The apparatus required :—**

1. Chemical balances with weights. Oertling's No. 3 will be found to be extremely suitable to all purposes. As shown in fig. 2, they consist of a twelve-inch beam which supports two pans, the ends of the beam being constructed

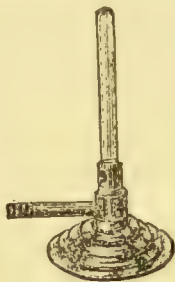


FIG. 1.—The Bunsen burner.

with straight knife edges upon which the pans are suspended by agate planes. The case is fitted with a sliding window in front, which can be closed and still admit of the scales being made to register by means of working a screw which projects externally. The balances must be kept in a dry room, away from any fireplace or door, and placed on a perfectly firm and level surface.

The operation of weighing consists of first lifting the beam off its support by means of the screw, and then noting by the long indicator which hangs down in front of the central vertical support of the balances whether the two pans exactly counterbalance each other; if not the balance must be adjusted by means of a small mechanism situated on the top of the centre of the cross-beam, and which can be moved horizontally to the right or left, according as it is necessary to increase the weight in either of these directions.

After thus seeing that the scales are accurately equipoised,



the material is then placed upon the tray to the operator's left, and the weights are added to the tray on the right.

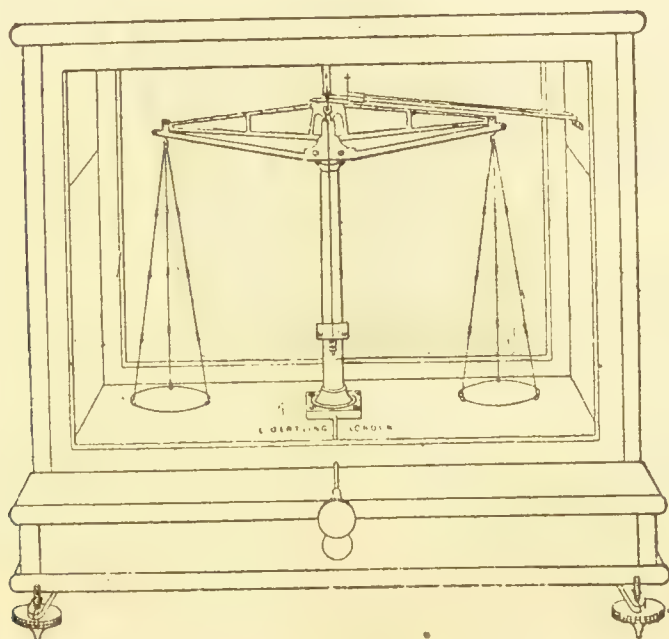


FIG. 2.—Chemical balances.

After the addition of each successive accretion of weight the result must, of course, be tested, but before any further addition or removal is made the scales must be brought to rest upon their supports, or the apparatus may be put out of gear.

Each of the weights is marked, as the plan of the box will best show (fig. 4). The larger brass weights (1—50) represent grammes, the next in size decigrammes (0.1—0.5), the next centigrammes (0.01—0.05), and small forceps are used for picking up and applying them to the pan. The milligrammes are added by a little piece of bent wire ("the rider"), which is carried by means of a sliding rod moving just above the level of one of the cross-beams, which latter

will be observed to show ten markings numbered from one to ten. By sliding the rod which supports the rider up to,

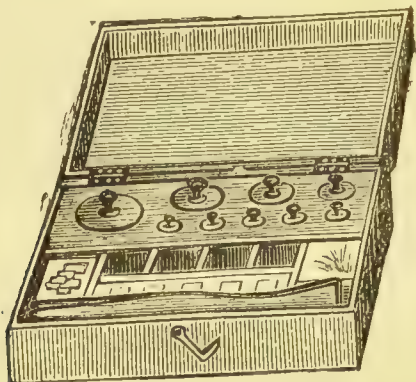


FIG. 3.—Box of chemical weights.

say, the marking No. 5, and then turning it round, the wire may be made to ride upon that number, and the carrier

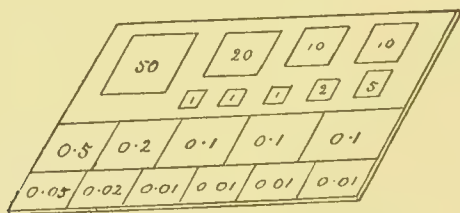


FIG. 4.—Plan showing the arrangement of the weights in box.

can subsequently be withdrawn ; five milligrammes of weight will then have been added to that side of the scales.

*Example.*—A small platinum dish is placed on that side of the scales which is not fitted with the apparatus for applying milligramme weights.

A five gramme weight is placed on the other pan.

The pans are lifted by means of a half turn of the screw, and the platinum dish is found to weigh down the five grammes.

The scales are put at rest, and a two gramme weight is

added to the five, and this is also carried up by the greater weight of the platinum dish.

Another gramme is added, and this is found to be too much, and is therefore removed, and a five decigramme weight (*i.e.*,  $\frac{1}{2}$  gramme) substituted.

The platinum dish is still slightly the heavier, therefore another decigramme is added, with the result that the weights now slightly overbalance the dish.

The one decigramme weight is therefore removed and a five centigramme substituted; not enough!

A two centigramme weight further added, however, so extremely nearly establishes the required equilibrium, that the addition of another centigramme over-reaches the mark.

Three milligrammes, added by means of the little "rider," are ultimately found necessary to effect such an uniformity between the weights of the contents of the two pans, that the index oscillates quite evenly on either side of the central mark in the porcelain, and would ultimately come to rest at that point.

The weight, therefore, of the platinum dish is:—

Seven grammes	=	7
Five decigrammes	=	0.5
Seven centigrammes	=	0.07
Three milligrammes	=	0.003
<hr/>		
Total	=	7.573

The dish need not be reweighed on every occasion of using, this is only necessary at intervals of every few days.

2. A platinum dish, capable of holding a little over 100 c.c. of water. Substances containing the heavy metals should not be ignited in a platinum dish.

3. Two shallow porcelain evaporating dishes, holding a little over 250 and 500 c.c. respectively (fig. 5).

4. Two porcelain crucibles with cover, for igniting residues (fig. 6).

5. Four white porcelain slabs, about six inches by four.

6. Four white porcelain basins of about five inches diameter (fig. 7).



FIG. 5.—Evaporating dish.



FIG. 6.—Crucible with lid.

7. A pestle and mortar (fig. 8).

8. Graham's or Liebig's condenser. Graham's will be found a most convenient instrument. As seen by fig. 28, it consists of a smaller glass tube, bent at one end, where it carries an indiarubber cork for attachment to a boiling flask. Surrounding this smaller tube for about three-fourths



FIG. 7.—Porcelain dish.



FIG. 8.—Pestle and mortar.

of its extent is a larger one, closed at both ends by indiarubber corks, the centres of which are perforated for the passage of the smaller tube. Cold water is made to circulate constantly in this outer tube by means of the tubing which connects its interior with the tap, the water escaping at the opposite end through the tubing which conducts to the sink. The cool water thus kept constantly circulating condenses the vapour in the inner tube, at the further extremity of which the condensed vapour finds its outlet and is collected.

9. A set ("nest") of glass beakers (fig. 9), and a few watch-glass covers for same.



## HYGIENIC ANALYSIS OF WATER.

10. A bell glass cover.

11. Six boiling-flasks or retorts (of about a litre capacity), one of which is seen attached to the condenser in fig. 28.

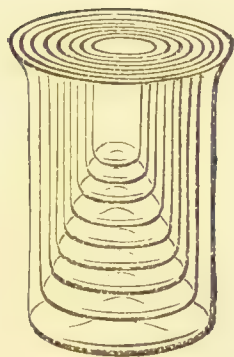


FIG. 9.—Nest of beakers.

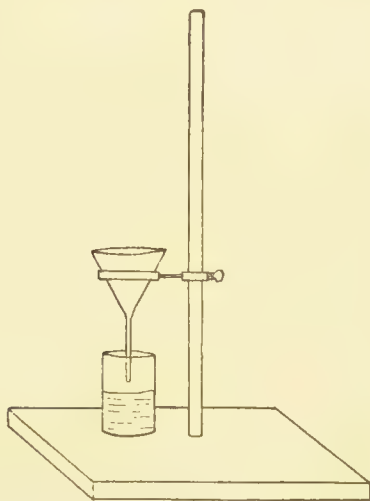


FIG. 10.—Filtering apparatus.

12. Glass stirring-rods.

13. A small glass-stoppered bottle (250 c.c.) for the soap test ; two (of about 400 c.c. capacity) for Tidy's process ; and one (of 250 c.c. capacity) for the estimation of oxidised nitrogen.

14. Glass funnels for filtration, with wooden support (fig. 10). The filter paper should never project beyond the funnel, and the fluid should be poured down a glass rod on to the filter.

15. Glass burettes holding ten cubic centimetres, and graduated in c.c.'s and one-tenths of c.c.'s, one of which should be mounted upon a wooden stand and should be stoppered at the top, and fitted with a stop-cock at the bottom (fig. 11).

In using an unmounted graduated burette it is always as well to fill it with the solution up to the highest mark

upon it, *i.e.*, in this case exactly 10 c.c. should be taken—even in those cases where very little of the solution is likely



FIG. 11.—Graduated burette.

to be required; otherwise, by the end of the process, the exact level to which the burette was charged may have been forgotten, and in consequence a repetition of the whole test becomes necessary at the sacrifice of time and material. When the delivery from the burette is controlled by the hand, it should be held vertically with the index finger guarding the upper extremity, and it is only after a little practice that the amount escaping from the lower extremity can be nicely controlled. When it is desired to regulate the escape in drops, the pressure of the index finger (which must be perfectly dry) upon the top of the burette is only gently relaxed—that is to say, almost imperceptibly lessened; otherwise the contents will escape too rapidly.

In judging the height to which fluid stands in a burette always take the level of the convex lower border of the meniscus which forms upon its upper surface, and make this rest upon the line to which the fluid is required to reach. Water standing to the level of 10 c.c. in a burette will appear, therefore, as in the accompanying figure (12).

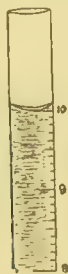


FIG. 12.—A burette filled up to the 10 c.c. mark.



FIG. 13.—Measuring flask.

The eye must always be on a level with the upper surface of the liquid when a reading is made. The burette just holds 10 c.c. of water, if at a temperature of about 60 deg. F., the water weighs 9.99 grammes. Similarly with a measuring flask (fig. 13), the graduation is correct if the 100 c.c. of water at about 60 deg. F. weigh 99.9 grammes.

16. Four graduated glass-flasks ("measuring flasks") marked respectively at the height to which 1,  $\frac{1}{2}$ ,  $\frac{1}{4}$  and  $\frac{1}{10}$  of a litre of water will stand. Fig. 13 shows one such flask.

17. Twelve Nessler glasses, four of which are shown in fig. 28.

18. A dozen test-tubes, with stand for same, and a test-tube cleaner and holder (fig. 14).

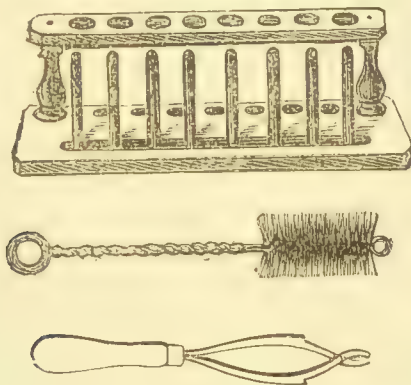


FIG. 14.—Test-tube stand, cleaner and holder.

19. A glass pipette. Pipettes should not be blown out, but allowed to drain, and the drop at the delivery end removed by touching the side of the vessel into which the contents of the pipette are emptied.

20. Two four-footed iron stands.

21. Two iron tripods (fig. 15).

22. Triangles of iron-wire lined with pipe-clay (fig. 16).

23. Stout fine wire gauze, cut about four inches square.

24. A pair of small crucible tongs (fig. 17), which may, with advantage, be platinum pointed.

25. A small copper water-bath, of about six inches diameter, and fitted with rings which adapt its mouth so as to

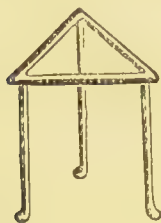


FIG. 15.—Iron tripod.



FIG. 16.

fit different sized evaporating dishes (fig. 18); or the water-bath recommended by the writer, which is more generally

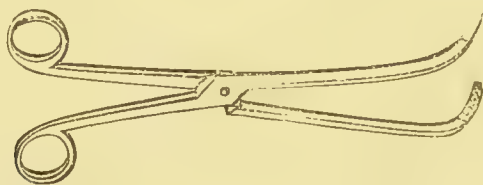


FIG. 17.—Crucible tongs.

serviceable in analyses other than those of water. This consists, as in fig. 19, of a water-bath of the common shape,



FIG. 18.—Ring water-bath.

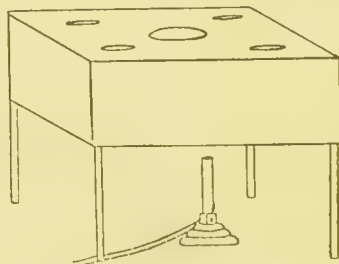


FIG. 19.—The form of water-bath recommended.

but in its roof are five openings, one large central and four small ones, at each corner. The diameter of the central opening is about  $2\frac{1}{2}$  inches, and that of each of the smaller ones about  $1\frac{1}{4}$  inches, and there must be a space of about  $1\frac{3}{4}$  inches from the margin of the central opening to the

margins of the others, so that there is room for dishes to stand over each of the apertures at the same time.

A water-bath is a receptacle which holds water, and admits of this water being heated up to a certain temperature ; when vessels containing liquids are made to stand in or over the water thus heated, evaporation of their fluid contents may be effected at any desirable temperature up to  $212^{\circ}$  deg. F. The heat thus applied must in any case be considerably below that which would be reached by the application of the naked flame, since it can never exceed that of the boiling point of water.

The apparatus shown in fig. 20 consists of a beaker partially



FIG. 20.—A water-bath improvised from an ordinary beaker.

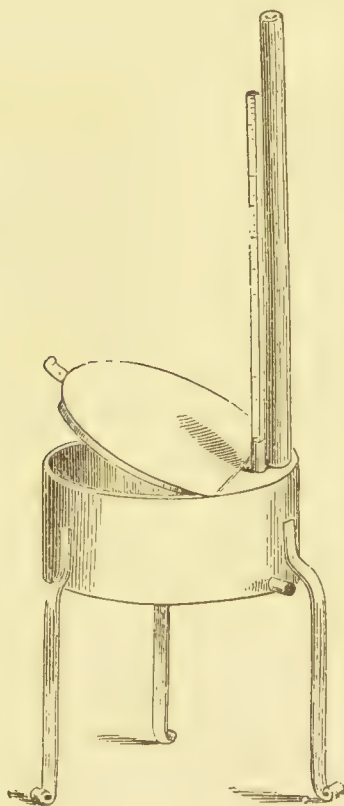


FIG. 21.—Taylor's hot-air-bath.



filled with water, on the top of which an evaporating basin is placed—provision being made for the escape of steam by a small piece of rolled blotting paper.

26. A hot air bath is of use when a higher temperature than that of 212 deg. F. is required for absolute drying. In fig. 21, a simple and cheap form (Taylor's) is shown, with the thermometer *in situ*, which registers the temperature of the interior of the oven. The temperature is regulated by the size and proximity of the flame that is applied to the under surface of the case, and by the degree to which the lid is opened. The object to be dried is placed inside the oven, upon a perforated tray, where it may be kept at any required temperature.

27. A packet of filter papers. Ordinary filter papers are liable to contain a trace of lime, magnesia, sesquioxide of iron, and other substances soluble in acids; where necessary, therefore, these substances should be removed by washing with acid. It is also necessary to have some specially prepared papers, of which the ash yielded by ignition is known for each paper. Papers are now sold which have been well treated with hydrochloric and hydrofluoric acids, and which yield an ash which is quite insignificant.

28. A long tube of thin colourless glass, known as "the two foot tube," employed in judging some of the physical characters of water.

29. A box of test papers, blue and reddened litmus and lead papers.

30. Two long thermometers graduated in Fahrenheit degrees.

31. Indiarubber corks (perforated and imperforated) to fit the boiling flasks.

32. A desiccator. A desiccator is simply a glass shade inside of which there is a vessel containing some agent which will keep the air free from moisture (such as strong sulphuric acid). A residue completely dried by heat will absorb a

little of the vapour from the atmosphere while cooling, and thus increase slightly in weight, unless the precaution is

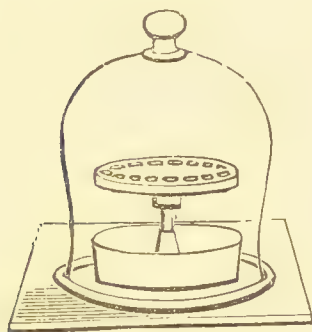


FIG. 22.—A desiccator.



FIG. 23.—A conical sediment glass.

taken to place it inside a desiccator during the cooling process. In the desiccator shown in fig. 22, the perforated tray supports the substance to be cooled, strong sulphuric acid is contained in the glass basin beneath, and the rim against which the cover closely fits is greased with tallow.

33. A conical sediment glass (fig. 23).

34. A wash bottle for the purpose of washing precipitates, etc. A fine jet of distilled water (or in some cases other agents more suitable for washing purposes) can be made to escape at the free opening of one of the glass tubes shown in fig. 24, by gently blowing down the other tube. The process of washing is completed when a drop of the last washing yields no residue when evaporated on a platinum spatula.

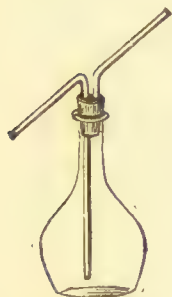


FIG. 24.—A wash-bottle.

35. Some stout platinum wire and a small platinum spatula.

36. Two special test-tubes, about one foot in length and one inch in diameter.

37. Apparatus for extracting gases from water, for Thresh's process, and for Marsh's process.

38. Hand lens and microscopic apparatus.

**The reagents required consist of:—**

1. Distilled water.
2. Distilled ammonia-free water.
3. Pure and dilute sulphuric, nitric, and hydrochloric acids.

4. Alkaline permanganate solution.

5. Nessler's reagent.

6. Pure phenol.

7. *Standard solutions of:—*

Chloride of ammonium.

Nitrate of silver.

Soap.

Nitrate of potassium.

Nitrite of potassium.

Chloride of calcium.

Permanganate of potassium.

Sulphate of copper.

Sulphate of zinc.

Acetate of lead.

Perchloride of iron.

Indigo.

Hyposulphite of sodium.

Decinormal sodium carbonate.

Decinormal hydrochloric acid.

8. *Solutions of:—*

Ammonia.

Chloride of ammonium.

Oxalate of ammonia.

Sulphide of ammonium.

Molybdate of ammonium.

Sulphuretted hydrogen.

Nitrate of silver.

Meta-phenylene-diamine.  
 Caustic potash.  
 Chromate of potassium.  
 Iodide of potassium.  
 Ferrocyanide of potassium.  
 Ferricyanide of potassium.  
 Cyanide of potassium.  
 Sulpho-cyanide of potassium.  
 Phosphate of sodium.  
 Nitro-prusside of sodium.  
 Chloride of sodium.  
 Hypo-chlorite of sodium.  
 Chloride of barium.  
 Brucine.  
 Starch.  
 Perchloride of mercury.  
 Methyl orange.  
 Phenol-phthalein.

9. Zinc foil and copper-turnings.  
 Granulated zinc.  
 Animal charcoal.

## TABLES OF ATOMIC WEIGHTS.

(By the later Researches).

Aluminium	(Al)	=	27.3
Arsenic	(As)	=	74.9
Barium	(Ba)	=	136.8
Bromine	(Br)	=	80
Calcium	(Ca)	=	39.9
Carbon	(C)	=	12.0
Chlorine	(Cl)	=	35.37
Chromium	(Cr)	=	52.4
Copper	(Cu)	=	63.18
Hydrogen	(H)	=	1.0
Iodine	(I)	=	126.5
Iron	(Fe)	=	55.9

Lead	(Pb)	=	206.4
Magnesium	(Mg)	=	23.94
Manganese	(Mn)	=	55.0
Nitrogen	(N)	=	14.0
Oxygen	(O)	=	16.0
Phosphorus	(P)	=	31.0
Potassium	(K)	=	39.0
Silver	(Ag)	=	107.7
Sodium	(Na)	=	23.0
Strontium	(Sr)	=	87.2
Sulphur	(S)	=	32.0
Tin	(Sn)	=	117.8
Zinc	(Zn)	=	64.9

### THE WEIGHTS AND MEASURES UPON THE METRICAL SYSTEM.

(A knowledge of which is necessary for water analysis).

#### Length.

The metrical system is founded upon the "metre," which is divided and multiplied by ten to represent different measures, as follows:—

1 millimetre	=	$\frac{1}{1000}$	part of a metre.
1 centimetre	=	$\frac{1}{100}$	" "
1 decimetre	=	$\frac{1}{10}$	" "
1 metre	=	39.37	inches.
1 decametre	=	10	metres.
1 hectometre	=	100	"
1 kilometre*	=	1000	"

#### Capacity.

1 cubic centimetre	=	0.061	cubic inches.
28.35 "	"	=	1 fluid ounce.
1000 "	"	=	1 cubic decimetre = 1 litre.
1000 litres	=	=	1 cubic metre.

\* The Latin prefix therefore indicates division, the Greek, multiplication.



*Weight.*

1 cubic centimetre of distilled water at 39deg. F., and 760 millimetres barometric pressure, weighs one *gramme*, which is the standard of weight.

1 milligramme	=	$\frac{1}{1000}$	part of a gramme.
1 centigramme	=	$\frac{1}{100}$	" "
1 decigramme	=	$\frac{1}{10}$	" "
1 gramme	=	15.432	grains.
1 decagramme	=	10	grammes.
1 hectogramme	=	100	"
1 kilogramme	=	1000	"
1 lb. avoirdupois	=	453.59	"
1 gallon of water	=	4.543487	litres.

The term "septem" is sometimes used. It simply implies seven *grains*.

*Thermometer Scales.*

Centigrade Freezing point	=	0	Boiling point	=	100
Réaumur "	=	0	" "	=	80
Fahrenheit "	=	32	" "	=	212
$\therefore \frac{\text{Centigrade}}{5} = \frac{\text{Réaumur}}{4} = \frac{\text{Fahrenheit}-32}{9}$					

∴ To convert Centigrade to Fahr.  $\times 9 \div 5$  and add 32.

" " Fahr. to Centigrade subtract 32 and  $\div 9 \times 5$ .

" " Réaumur to Fahr.  $\div 4 \times 9$  and add 32.

A VOLUMETRIC ANALYSIS signifies an analysis by measure.

A GRAVIMETRIC ANALYSIS signifies an analysis by weight.

## CHAPTER II.

## A WATER REPORT.

For hygienic purposes such a report must include all the information which can be obtained regarding the extent to which organic matter at present exists ; the evidence of former pollution by this material in the oxidised, etc., products of its decomposition ; and the presence of harmful saline material and poisonous metals.

Much unnecessary confusion is created by the fact that no fixed terms are universally recognised and adopted by which results may be always expressed. The results in the several steps of the analysis are, in consequence, variously returned by different analysts in terms of :—

Parts per 100.

Grains per gallon.

Parts per 100,000.

Parts per 1,000,000.

Parts per 100,000,000.

It seems most desirable, therefore, that some uniformity should be established upon this point. Undoubtedly the best amount for working purposes in many of the stages of the analysis is 100 c.c. of the water, and the result, if this quantity be taken, can generally be at once expressed in "parts per 100,000" (since 100 c.c. = 100,000 milligrammes) ; this is the most common return made in this country, and it is moreover in general use in France and Germany, etc.

Such then will be the quantitative expression used throughout Part I. of this book, but as the poisonous metals are so

generally expressed in terms of grains per gallon, an exception is made with regard to these. Where, however, the terms employed are other than those to which the reader has grown familiar, he should convert them into such before attempting to form an opinion of the water.

Supposing a report reads "chlorine 2.8 grains per gallon;" how many parts per 100,000 will this represent?

Now there are 70,000 grains in a gallon.

∴ „ „ 2.8 grains in 70,000 grains or 2.8 parts per 70,000 parts.

∴ as 70,000 : 100,000 :: 2.8 :  $x$  (parts per 100,000).

Or as 7 : 10 :: 2.8 :  $x = 4$  (parts per 100,000).

It is thus seen that it is only necessary to multiply results returned in "grains per gallon" by 10, and to divide by 7, in order to convert them into "parts per 100,000," since grains per gallon are parts per 70,000. The converse of this also applies, of course, and if it be desired to convert "parts per 100,000" to "grains per gallon," the returns must be multiplied by 7 and divided by 10.

Where the results of a quantitative test are desired to be returned in terms of "grains per gallon," it is convenient to measure out 70 c.c. of the sample, and to work with this. The reason for this is, that 70 c.c. represent "a miniature gallon" so-called, and the results can at once be expressed in terms of an imperial gallon. The relation between the so-called "miniature gallon" and the imperial gallon depends upon the following facts:—

1 c.c. of water is taken to weigh one gramme, at the usual temperature of the laboratory.

Therefore 70 c.c. ("in the miniature gallon") of water weigh 70 grammes or 70,000 milligrammes.

Therefore since there are 70,000 component parts in either case (of milligrammes in one, and grains in the other), the 70,000 milligrammes may be taken to represent "a miniature

gallon," in which the 70,000 grains of the imperial measure are represented by milligrammes. The results arrived at in milligrammes, where 70 c.c. of water are taken, can therefore be at once expressed in terms of "grains per gallon."

Subjoined is a copy of the Report now in use at the Hygienic Laboratory, University College.

The Hygienic Laboratory,  
University College, W.C.

Report on Analysis of                      sample of water received on  
from

Name or number of Sample . . . . .	
Date of Collection . . . . .	
Physical Characters . . . . .	
Reaction . . . . .	
Saline (or "Free") Ammonia . . . . .	} Parts per hundred thou- sand.
Organic (or "Albuminoid") Ammonia . . . . .	
Oxygen absorbed from Permanganate in one hour at 80° F. . . . .	
Total solid matters . . . . .	
(a) Volatile . . . . .	} Parts per hundred thou- sand.
(b) Fixed . . . . .	
(c) Appearance on ignition . . . . .	
Total Hardness . . . . .	} Parts per hundred thou- sand.
(a) Temporary . . . . .	
(b) Permanent . . . . .	
Chlorine . . . . .	} Parts per hundred thou- sand.
Equivalent to common salt. . . . .	
Nitrogen as Nitrates and Nitrites . . . . .	} Grains per gallon.
Poisonous metals . . . . .	
Nitrites . . . . .	
Phosphates . . . . .	
Sulphates . . . . .	
Microscopical Examination of the Sediment. . . . .	

Remarks

Date of examination.

Signed

## CHAPTER III.

## THE COLLECTION OF SAMPLES.

SINCE the analyst will be frequently asked for instructions concerning the mode of collecting a sample of drinking water for analysis, it will be opportune to consider at the outset a few points under this head.

At first sight it appears that the matter may be dismissed off-hand by a simple statement to the effect that such water should always be collected for analysis just as it is ordinarily obtained for drinking purposes, and such is broadly correct. It is obvious, since our object is to discover the possibilities of danger in addition to actual danger present, we should not effect this by collecting the water under circumstances which would show the least amount of dangerous pollution; but rather, on the other hand, an endeavour should be made to ascertain the maximum amount of pollution to which the water is liable, in order that this may be taken as a gauge of its potentialities for evil. Instead, then, if the collection be made from a house-tap, of allowing the water to run for some time before taking the sample, as is so generally recommended, it should be a safer and better policy—if there is nothing to show that some of the water is always first run off and discarded by the consumers—to include it in the sample taken. It must be borne in mind, however, in drawing one's conclusions, that the water which has been standing overnight in the pipes, is very liable to show traces of lead, whereas the rest of the water drawn throughout the day, may be quite free from this metal.

In the case of streams, lakes, etc., the entrance of any source



of pollution, floating scum, etc., should only be avoided to the same extent as it is by those who come to collect their drinking water, and all the conditions of such collection should be closely imitated in taking the sample.

When there is a general system of water supply, an effort must be made to meet the same ends by choosing samples from the street fountains and street mains, rather than from storage, etc., reservoirs, and the reputation of the water supply must rest upon the result of an analysis of such samples. Since, moreover, impurities may be added in its storage and distribution about the house, it would not be fair in all cases to judge a public supply from the tap water of any particular dwelling.

Following out the principles advocated, with regard to those wells from which the water is removed by pumping, it is advisable to continue the process for some time, but not longer than it is judged the water might be pumped during any one day under the prevailing circumstances of demand.

This is done because the last "pumpings" will often yield the greatest evidence of pollution. The detection of a very fine amount of organic contamination may often be made by collecting samples from the wells of any district, and taking the purest of these waters as a standard for comparison with the others.

When, however, the fact is borne in mind that the water from many wells is so materially influenced both as to quantity and quality by the rainfall, it will be understood how samples from the same well may vary in purity according as a long dry period may have preceded the collection, or a heavy rain-downpour—which may be the means of conveying to the well water impregnated with surface washings, or water which may have dissolved the accumulated filth out of the pores of the soil. These facts should always be well weighed, at least in those cases where the well is not a very

deep one, and is not carefully covered and well coped ; and a further sample should be requested at a time when the well has run the maximum risk of pollution.

The fact as to whether a cesspool, etc., drains into a well, can readily be decided by either introducing a considerable quantity of sodium chloride into the cesspool, followed by plenty of water, and estimating the chlorine in the well water from 24 to 48 hours afterwards, or by introducing aniline red or prussian blue into the cesspool, and endeavouring to detect the colour in the water.

Water is customarily collected for analysis in a large dark blue or pale green glass-stoppered bottle, called "a Winchester quart," which holds, however, about twice the amount which is implied in its name, *i.e.*, about half a gallon. Stout wicker covers are made to protect them in transit by parcel post or rail. These bottles have become generally adopted because, in addition to holding an amount which meets all the requirements of an ordinary analysis (even though it be necessary to repeat some of the tests), they are strongly made and of a convenient shape ; but obviously any stout glass bottle of pretty much the same dimensions, fitted with a glass stopper, will serve the same end. Where a mineral analysis is required it is necessary to have quite two gallons of the water. It is well to avoid the employment of stoneware bottles.

The bottle must be thoroughly cleansed by first well rinsing with a little dilute hydrochloric acid, and then well washing in good water until the washings are no longer acid.

The sample is then thus collected and dealt with :—The bottle is first quite filled with the water, and then emptied ; it is again completely filled up (in a manner which will not favour the aeration of the water), and the glass stopper, having been found to fit accurately and tightly, is tied down firmly on to the neck of the bottle, and the knots are protected

with sealing wax. A label is then attached, care is taken to keep the sample cool and unexposed to light until the analysis is commenced, and under no circumstances should the major part of the analysis be delayed more than 48 hours, or important chemical changes may transpire, *i.e.*, organic matter may suffer a very slight reduction, free ammonia may increase or decrease in amount, nitrates may be reduced, calcium or magnesium carbonates and iron, which were held in solution by carbonic acid, may, owing to the escape of the carbonic acid, be partially deposited.



FIG. 25.—The Winchester quart bottle with label affixed.



FIG. 26.—Wicker case for the Winchester quart bottle.

Although information should be generally demanded on all the points bearing upon the possibilities of pollution which the water has incurred, it is often best not to avail oneself of the information until the analysis is completed, otherwise

there is a strong incentive to treat cursorily some part of the analysis, on the supposition that it is unnecessary, which with due care might have disclosed an undreamt of source of pollution. Regarding such information, that bearing upon the constitution of the strata through or over which the water has passed is most important to the analyst, since certain ingredients are only in default of such a source indicative of organic pollution. Any one is able to furnish information as to whether the soil consists of such familiar substances as clay, gravel, sand, chalk, or vegetable mould, and whether the subsoil, exposed as it is by railway or road cuttings, is of chalk, sandstone, etc.

It is necessary to keep in view the fact that our work is essentially in the cause of disease prevention, and that our duties and responsibilities do not end in returning an analytical report; and that, as sanitarians, all information as regards the risks of pollution are of great value as indicating possibilities of danger, when such dangers may not be made manifest at the time in an analysis; it is also of great importance to know, in every instance, the motive for requiring an analysis, as one may often thus learn the presence of some preventable disease in a household, which is not necessarily connected with the water, though attributed to it.

It seems hardly necessary to point out that the lesser degrees of pollution are sometimes worthy of our consideration. A water may effect harm without creating manifest sickness, and many obscure conditions of slightly impaired health and vigour may be due to a slightly impure water supply, such as one would not, under ordinary circumstances, feel justified in condemning.

It is often difficult to form a correct opinion of a sample as to its purity or its potentiality for danger without the knowledge of some of the circumstances of its collection, and if the water is held to run great risk, this should suffice for its con-



demnation, although the chemical analysis at the time might prove satisfactory; for a little filtration through earth may suffice to purify a water for a time, but there is always a possibility of the purifying powers being exhausted at any moment, and the danger from the consumption of the water is a constant one. It is very desirable that labels should be given to those collecting samples, and that these are stuck upon the bottle in each case. The subjoined label, when filled in, would convey all necessary information to the analyst.

SAMPLE OF WATER FOR ANALYSIS.

Reason for desiring an analysis

Date of collection

Source and method of collection

Geological characters of the soil and subsoil of the district

The rainfall during the previous week (*i.e.*, in such terms as "nil"  
"small" or "great" in amount)

Nature and distance of any evident source of pollution.

The result of every analysis should be carefully entered into a book kept for the purpose, and such a record becomes most valuable for making comparisons with future samples of water from the same sources.



## CHAPTER IV.

## THE ANALYSIS.

It has been said, and truly so, that so small an amount of organic matter as would not call for condemnation in the water, may yet contain the specific germs of disease, and that therefore it is only from the results of a bacteriological, combined with a chemical examination, that a water may ever be classified as *safe*. At present, however, there are so many difficulties to surmount in the separation and positive identification of specific bacteria in water, that a biological examination cannot replace a chemical one, and in the main a chemical analysis may be held to be very reliable; for it is logical to contend—and experience supports the contention—that organic matter being the food pabulum for specific micro-organisms in water, the probability of their presence should bear a direct ratio to the amount of such organic matter present; and that, moreover, the germs of typhoid and cholera always gains access to the water in their natural media of fæcal matter and filth, and it is extremely rare that this escapes detection by the chemical tests usually employed.

At the very outset it must be realised that in forming an opinion as to whether a water is fit for human drink, organic matter (since an analysis mainly aims at detecting this) will give evidence of its presence in most of the steps which form a complete analysis, and that it is this collective evidence which determines the opinion to be formed, and not the evidence which any one special test may offer. This point is well illustrated in the physical characters of water, for whereas polluted shallow well waters are notoriously often clear, spark-

ling, and pleasant to the palate (though affording direct evidence of organic pollution "all along the line" of the analysis), yet these characters are precisely those of our purest and best waters, so that the result of the physical examination alone would be very misleading.

For these reasons, and from what follows, it will be seen that evidence of purity or pollution furnished by the senses, is very unreliable in itself, and is not even of much corroborative value. Such tests, therefore, are not worthy of lengthy consideration here.

### THE PHYSICAL CHARACTERS.

The sample of water should be well shaken, and then about a litre (1000 c.c.) is emptied into a glass beaker, which is kept covered so as to protect from suspended matters in the air, and after the bulk of any suspended matter which may be present has settled, a thin colourless glass tube, twenty-four inches long, is filled with the clear (supernatant) water, and from the appearance of this in the "two foot tube," as it is called, the following physical characters are judged:—

1. **Clearness.**—Though the best waters are always bright and clear, these qualities cannot be considered as evidence of purity, for a polluted well-water may also possess them; and on the other hand, any slight haziness or turbidity—which is of course furnished by minute particles of suspended matter—may by chemical and microscopical examination be proved either innocuous or harmful. The degrees of any such turbidity may be expressed as "very slightly turbid," "slightly turbid," and "turbid."

2. **Colour.**—To detect this the tube is fixed vertically upon a white porcelain slab, and the observer looks down through the depth of the column of water on to the slab, which forms the background. It is only in this manner that the faintest degrees of coloration are best appreciated, if

even they be detected at all; and when thus examined it is rare that the water is not seen to possess some colour, however faint. In a good water it is generally of an extremely faint greyish-blue or greenish tint, and if there is any doubt upon this point another similar tube should be filled with colourless distilled water, and a comparison made after the two waters have been placed under exactly the same conditions of light access, etc. Filtration of the water would show whether the colour is due to suspended or dissolved matter.

The various hues of yellow and brown will denote the presence of either animal or vegetable pollution (*i.e.*, sewage or peat), or mineral contamination such as iron or clay—but if such colour is due to iron or clay, a sediment will form, consisting, in the case of iron, of the hydrated ferric oxide (*i.e.*, “rust”).

It follows from what has already been seen that the amount of sewage pollution must be very high to furnish any colour.

A marked green denotes the presence of vegetable matter containing chlorophyll, which will generally be found to mainly consist of the harmless algæ. The water in the neighbourhood of dye-works may be of course variously coloured.

Colour alone is thus seen to afford no justification for condemning a water until the nature of the material creating it is known; peat, for example, present to quite a harmless extent, will often colour a water markedly. The importance of the test, *per se*, does not seem to warrant any attempt at definite measurement, but if such is desired it is best performed by the method of Crookes, Odling, and Tidy.

An empty tube, exactly similar in every respect to that containing the water to be compared, is employed; this has two hollow glass wedges behind it, the one filled with a  $\frac{1}{2}$  per cent. sulphate of copper solution, and the other with a mixture of ferric chloride (0.7 gramme per litre) and cobalt chloride (0.3 gramme per litre) solutions with a very slight excess of

hydrochloric acid. These wedges are made to slide across one another in front of a circular aperture in a metal sheet, and thus any desired combination of brown and blue can be obtained. They are pushed over the empty tube until the colour, on looking down it, appears to be identical with that of the water. Each prism is graduated from 1 to 50, the figures indicating millimetres in depth of the solution at that particular part of the prism, and the degree of colour is expressed as equivalent to so many millimetres of blue, and so many of brown solution.

**3. Taste.**—The pleasant taste of good water is furnished by the gases dissolved in it, but since water must contain large quantities of any ingredient for its presence to be detected by the sense of taste, as an indication of dangerous contamination, the test is, with one exception, useless; for among the many impurities to which ordinary drinking water is liable, iron stands alone as offering any valuable indication of its presence by this means; so little as  $\frac{1}{4}$  grain to the gallon, of this metal, will impart a chalybeate flavour. Chloride of sodium (common salt) may be present in enormous quantities (75 grains to the gallon) without causing a brackish taste, and waters foully polluted with organic matter are often so palatable that wells, etc., containing them are frequently patronised by the public in preference to those which contain much purer waters.

It is not advisable in every case to taste samples sent for analysis, and the analyst must exercise his discretion from the information which he receives with the sample as to the safety of such a procedure.

**4. Smell.**—This is best detected by placing about 150 c.c. of the sample into a 250 c.c. glass stoppered bottle (itself odourless), almost completely immersing this in hot water at about 140 deg. F. for a few minutes, and then taking prolonged (deep) sniffs at its contents after removing the stopper. For most practical purposes, however, it will suffice if the

sample is smelt after it has been thoroughly shaken ; and it is only necessary when a suspicion remains after this procedure to resort to the plan of heating the water—except it be in those cases where there is strong reason for considering that odoriferous gases may be present in small quantities inappreciable except by heat, such, for instance, as would arise when the water is judged to have run risk of coal-gas contamination, etc. The addition to the water of a little strong potassic hydrate solution helps to make the recognition of such gases more easy.

The test of smell is unreliable, and none may be evident in waters which are considerably polluted by sewage ; it must be borne in mind also that many of the noxious materials which may gain access to a water, have little, if any, smell originally.

Any coal gas, sulphuretted hydrogen, or ammonium sulphide present, would thus be detected, and more especially if the water is heated to about 120 deg. F.

The variety of odours which may be given off from a water defies description ; many of them, though quite peculiar and distinct, it seems quite impossible to describe, and any comparisons made with more familiar smells frequently only appeal to a certain proportion of those who test them.

A distinctly putrid odour is characteristic of large quantities of decomposing animal or vegetable matter, and a *urinous* odour is sometimes distinctly perceptible when fresh sewage has gained access to the water. The rotten egg smell of sulphuretted hydrogen and that of coal gas are both peculiar and distinctive. The presence of any of these odours would condemn the water, as indeed should any other if well marked (*vide* "Gases and Vapours in Water").

It seems unnecessary to define the less distinctive odours, unless these can give some clue as to the nature of the pollution. Under any circumstances it is far better that the analyst describes the odour in his own words than he should



be cramped by any desire to confine his returns within the category of any such terms as "musty," "horse-pond-like," "pig-odour," "fishy," and "cucumber-like," etc.

Sulphuretted hydrogen may mask other odours; the addition of a little copper sulphate solution will prevent this.

5. **Aeration.**—Evidence of this is afforded by minute air bubbles collecting at the sides and bottom of the tube, and rising up occasionally through the water to the surface, and also by the degree of lustre the water possesses. The best waters are invariably well aerated; those from the chalk and limestone formations contain much carbonic acid, and little or none of any other gas, and where the water from these strata has been subjected to conditions of very high pressure and low temperature, carbonic acid may even be in such quantities as to give a turbid appearance to the water; when this is first exposed to the air.

The degree of aeration is of no value by itself, in the estimation of the purity or impurity of a water, though a good water, to be palatable, must be well aerated (*vide* "Gases and Vapours in Water"). Many deep well and spring waters, of great purity, are poorly aerated, and many foul waters are particularly bright and sparkling (chiefly from carbonic acid).

6. **Reaction.**—This is important, not so much as affecting an opinion upon the wholesomeness of the water, as from the value the knowledge acquires in some further stages in the analysis, for the correct performance of which it becomes necessary to neutralise any acidity in the sample. Though the most polluted waters from animal organic matter are generally decidedly alkaline from the carbonate of ammonia furnished by urine decomposition, yet most waters will be found to be faintly alkaline, the alkalinity being generally given by calcium carbonate, and less often by sodium carbonate, etc.

Some waters will be found to be neutral in reaction, and

acid waters are by no means uncommon in this country ; the latter most frequently gain their acidity from the peaty acids (humic, ulmic, and geic) taken up from the vegetable matter encountered in their surface flow, and such acidity is a marked feature of all so-called " peaty waters."

The test may be made by partially immersing in the water pieces of delicate blue and red litmus papers, and noting the change after a few minutes.

If the water gives an acid reaction it should be boiled, allowed to cool, and tested again. If the acidity has been lost it was due to free carbonic acid. Free acidity in water is generally due to carbonic acid, but may result also from sulphuric acid or organic acids. The sulphuric acid may gain access to the water from the oxidation of the iron pyrites in the soil, or, like other acids, from waste of factories. Alkalinity that disappears on boiling is due to free ammonia.

It is not generally necessary for hygienic purposes to test the *degree* of alkalinity or acidity of the water, but it is sometimes useful to make such an estimation, and the method is set forth in Chapter XVII.

7. **The Sediment.**—The presence of this, together with its macroscopic appearance, should be noted at this stage, but any opinion of its nature must be reserved until a microscopic and chemical examination has been made. Certain waters deposit calcium carbonate and iron when allowed to stand exposed to the air ; this is due to the escape of the carbonic acid that held these substances in solution.

## CHAPTER V.

## CHLORINE.

CHLORINE exists in most waters as chloride of sodium, and less frequently as potassium or calcium chloride. Free chlorine is rarely found in waters polluted by industrial waste products.

Combined chlorine is present in all waters to a small extent, even in rain water, since all atmospheres contain sodium chloride in suspension. It is only when its presence is marked that suspicion is aroused as to its origin.

The chlorine in rain-water varies from 0.25—0.5 pt. per 100,000 and in upland surface waters it is generally about 1 pt. per 100,000.

The presence of an excess of chlorides in water is due to either of the following causes:—

- (a) Deep wells by the sea coast; or those in which the water has previously percolated strata into the composition of which saline constituents enter, *i.e.*, greensand, sandstone, the London clay, and to a less extent the chalk. In the districts of salt deposits the water may contain very large quantities of chlorides.
- (b) Pollution by animal organic matter, and chiefly urine.
- (c) A mixture with sea water, as in tidal rivers.
- (d) Open reservoirs and other expanses of fresh water stored near the coast, also show slight excess—due to the fact that the atmosphere contains larger amounts of sodium chloride than is usual elsewhere,

and this in part gets deposited upon the surface of such waters.

(e) Effluents from alkali, and other industrial works ; salt, mines, etc.

The presence of chlorine in excess is thus seen—after excluding any possibilities the water may have run in acquiring its chlorine from sources (a) (c) (d) and (e)—to indicate organic contamination, and that of an animal nature, since vegetable pollution *per se* furnishes no such excess ; hence in these cases the estimation affords most important evidence as to whether a vitiated water is polluted by animal or by vegetable matter. Although the presence of but little chlorine is pretty conclusive evidence of the water's purity as regards animal pollution, it could not under ordinary circumstances be used as a rapid means of gauging the general purity of a water, since such may be injuriously contaminated with vegetable matter, or even with animal effluvia, and give no evidence of these in any excess of chlorine. When, however, the amount of chlorine which the purest waters in the district contain is known, any one water sample from the same district may generally be judged—as regards the presence of animal pollution—by estimating the chlorine alone, and noting if this is excessive.

It remains now to be seen, apart from any other information which has been furnished or which is available, what *chemical* means there are of excluding causes (a) (c) (d) and (e), as furnishing excess of chlorine. We are so generally dependent upon water which has come in contact somewhere in its course with the chalk and sandstone formations in England, that the difficulty which will probably most often arise is to decide as to whether a little excess of chlorine is not due to the fact that the water has been collected from these strata—which furnish soluble chlorides ; rather than to the presence of animal pollution. The matter fortunately becomes an easy one

to decide upon, when it is borne in mind that if the excess be derived from the strata, the total solids will be *high, and apparently, when ignited, entirely of a mineral nature*, and there will be no evidence of organic pollution furnished by those other stages of the analysis which aim at detecting such pollution. Thus waters from the greensand will yield a large amount of chlorine, but will generally furnish evidence of a high state of purity as regards organic matter.

If, on the other hand, the excess be due to animal pollution (it cannot be to vegetable), the total solid residue, though it may be high, will contain much organic matter, and there will be further evidence of the nature of the contamination all along the line of those steps of the analysis which serve to indicate such pollution. The amount of chlorine originally present is but little affected by subsequent filtration of the water through soil or strata.

If a large amount of chlorine be due to admixture with sea water, there will also be present large quantities of magnesium salts, which fact enables a decision at once to be made upon this score.

#### QUALITATIVE TEST.

The presence of combined chlorine (as chlorides) may be best detected by the addition of a few drops of a solution of silver nitrate and dilute nitric acid to the water in a test-tube, when a white haze, turbidity, or precipitate of argentic chloride will appear, according to the amount of chlorine present.

#### QUANTITATIVE TEST.

**Apparatus required.**—This is of the simplest nature:—

1. A burette graduated in tenths of cubic centimetres.
2. A white porcelain basin capable of holding about 200 c.c. of water.



3. A glass stirring rod.
4. A measuring flask for 100 c.c. of water.

**Chemical reagents :—**

1. A cold saturated solution of the yellow chromate of potassium, which, since it is to be added to the water, must obviously be free from chlorine ; this may be proved by acidulating a little of the solution by dilute nitric acid, and then adding a drop of nitrate of silver solution ; in the absence of chlorine the solution will remain absolutely clear.

2. A standard solution of silver nitrate, made to the strength that 1 c.c. is capable of precipitating 1 milligramme of chlorine ; made by adding 4.798 grammes of pure recrystallised silver nitrate in a litre of distilled water.

**THE PROCESS.**

1. Measure out 100 c.c. of the water and place it in the white porcelain dish.

2. Add a few drops of the solution of yellow chromate of potassium until a distinct yellow colour is furnished to the water. The object in adding this reagent is to make it serve as an "indicator," which shall denote at once the stage when all the chlorine has been precipitated.

3. The burette is charged with the standard solution of nitrate of silver, and this is added drop by drop to the water, when a dull reddish precipitate is at once formed of the red silver chromate ; this when stirred up in the water by means of the glass rod, at once disappears, owing to the chlorine in the water displacing the chromic acid, and itself combining with the silver, to form a white precipitate of the chloride of silver. As the addition of the standard solution is continued, the water, though it retains the yellow colour, becomes very turbid, owing to the accumulation of this precipitate of silver chloride ; and at length a point is reached, at which, there being no longer any chlorine left which is not

already combined with the silver, the chromic acid holds undisputed possession of this metal, and the red silver chromate remains permanently present; the first evidence of this is afforded by the yellow colour changing into an orange. After further addition of the silver salt, the whole solution becomes a dull red.

The motive for originally adding the "indicator," *i.e.*, the yellow chromate of potassium, becomes very evident by this stage, since there would otherwise be no means of knowing when just the required amount of silver nitrate had been added; for it would be impossible to judge of the exact stage when the maximum amount of white precipitate of silver chloride had been created.

4. The first evidence of any red colour remaining permanent, so as to furnish with the original yellow an orange colour, should be the clue for withholding any further addition of the silver nitrate—or the amount of chlorine, estimated as it is from the amount of the solution of the silver salt used, will be over-estimated.

5. It is then seen by the burette how much silver nitrate solution has been added, and each c.c. of this will represent 1 milligramme of chlorine.

*Example.*—5 c.c. of the standard solution of silver nitrate were required to use up all the chlorine in 100 c.c. of water.

But 1 c.c. of the solution = 1 milligramme of chlorine

∴ 5 c.c.    "        "        = 5        "        "

∴ there are 5 milligrammes of chlorine in 100 c.c. of water.

But 100 c.c. of water = 100 grammes = 100,000 milligrammes.

∴ there are 5 milligrammes of chlorine in 100,000 milligrammes of water, or 5 *parts per* 100,000.

*Conclusions to be drawn from the amount estimated.*—In a water collected from the chalk or red sandstone there is no

occasion to grow suspicious of the presence of chlorine until it reaches the proportion of 3 parts per 100,000. It must always be borne in mind, however, that its presence in excess of this amount can only be attributed to animal organic pollution after the likelihood of certain other sources having furnished it have been excluded. Deep well waters in green-sand strata may yield as much as 15 parts per 100,000 in cases where the water is absolutely free from animal pollution. Up-land surface waters free from animal pollution rarely contain more than 1 part per 100,000.

#### NOTES UPON THE PROCESS.

It is highly important that neither the water nor the standard solution of silver nitrate should be acid, or the results are incorrect—since the red silver chromate, being soluble in an acid medium, is dissolved, and does not remain to furnish the orange colour at the precise stage when all the chlorine has been exhausted. In these cases the smallest quantity necessary of precipitated calcium carbonate should be added to effect neutrality.

In estimating small quantities of chlorine it is advisable to first concentrate the water before titrating, as the results are otherwise very slightly in excess. Results are never absolutely correct if traces of alkaline silicates are present in the water, and the presence of nitrates and phosphates appears to affect the estimation. For those who have not a keen appreciation of colour change, it is desirable to prepare a second dish of water, to which a similar amount of the chromate has been added ; if this is placed alongside the water under examination it serves as a comparison whereby to judge the commencement of the colour change.

The chlorine is sometimes—in order that its amount shall be more readily appreciated by the laity—expressed in terms of the common salt it is equivalent to. This is readily

calculated by a comparison between the atomic weight of chlorine and the molecular weight of NaCl (common salt).

The atomic weight of chlorine is 35.37, and that of sodium is 23;  $\therefore \text{NaCl} = (35.37 + 23) = 58.37$ .

$$\therefore \text{Cl} = \frac{35.37}{58.37} \text{ of NaCl.}$$

Now the chlorine in the example taken amounted to 5 parts per 100,000; let  $x$  = the amount of NaCl this is

equivalent to; then  $5 = \frac{35.37}{58.37} x$

$$\text{or } x = \frac{58.37 \times 5}{35.37} = 8.25$$

$\therefore$  there would be 8.25 parts of NaCl per 100,000; if all the chlorine present were furnished by sodium chloride.

Thus the weight of chlorine  $\times \frac{58.37}{35.37} = 1.65 =$  the weight of sodium chloride.

## CHAPTER VI.

## HARDNESS.

THE estimation of "hardness" is more of economic concern than of hygienic, and its main importance is to decide whether the amount of lime salts (which chiefly create the hardness) is such as to render the water unsuitable for washing and cooking purposes, and for some trade processes. A hard water entails in its use a great waste of soap and considerable difficulty in procuring a lather, and when used for personal ablution is often found deleterious to delicate skins; it does not extract the same amount of strength from coffee, tea-leaves, and meat substances used for making soups, stews and gravies, as softer water; and vegetables boiled in it lose much of their flavour and colour. On the other hand moderately hard waters are always more palatable than the very soft ones.

It must not be thought, however, that "hardness" in a water is a factor which can be altogether disregarded from a health standpoint, for gastro-intestinal derangement, of a nature varying with the constitution of the salts which form the "hardness," may arise from the constant ingestion of a hard water among those constitutionally susceptible. As will be seen, the "permanent hardness" is generally mainly due to sulphates of the alkaline earths, and these have an aperient action, so that when they exist in large amounts the water is recommended and used medicinally on account of this property. Epsom, Leamington, Scarborough, and Cheltenham provide such waters.

Finally, hard waters, from the deposit they yield to boilers, are more apt to be the cause of explosions in these receptacles than other waters.

The order in which the salts are deposited is the following:—

The carbonates of calcium and magnesium.

The sulphates of calcium and magnesium.

Salts of iron, as bases or oxides, when present.

The silica or alumina, when present.

It is calculated that  $\frac{1}{4}$  inch of the incrustation—which is a bad conductor of heat—causes the waste of 45 per cent. of coal.

For trade processes generally—apart from the waste of fuel and danger occasioned by the “crust” which hard waters yield to boilers—it is of great importance to the process itself that the water should be moderately soft.

The factors which commonly cause “hardness” in a water are the following:—

Calcium and magnesium salts (*i.e.*, carbonates, sulphates, phosphates, chlorides, and nitrates).

Free carbonic acid in the water, or free mineral or vegetable acids.

Iron, silica, and alumina.

“The total hardness” in most of the drinking waters of this country is largely furnished by calcium and magnesium salts, and free carbonic acid; and more especially is such hardness due to calcium salts. Waters, therefore, from the chalk, oolite, limestone, dolomite, and new red sandstone formations—which strata consist largely of various proportions of these several salts of calcium and magnesium—will be those which furnish the highest degrees of hardness.

Of the calcium and magnesium salts, the carbonates very greatly predominate as the cause of “hardness.” The carbonates of calcium and magnesium are almost insoluble in



pure water ; but they are held in solution by carbonic acid—in the form of bicarbonates.

Assuming a water to have its "total hardness" built up of all the materials above enumerated, then if it be well boiled some of these become precipitated, and being no longer in solution, they cease, of course, to add to the "total hardness"—the amount thus lost is accordingly termed "temporary hardness," and that remaining "permanent hardness."

Of what does this "temporary hardness" consist?—or in other words what materials are precipitated by the boiling? Foremost in importance is the fact that the carbonic acid which held the carbonates of calcium and magnesium in solution is driven off, so that these salts, no longer soluble, fall to the bottom. Any other constituent which was held in solution by the free carbonic acid present, such as iron, is also precipitated (iron, in the form of the hydrated ferric oxide). Phosphate of lime, silica, and the sulphate of lime, if present in large quantity, would also be in part precipitated.

The "permanent hardness," then, consists of what still remains in solution, *i.e.*, calcium and magnesium sulphates, phosphates, chlorides and nitrates, and maybe a little iron, silica, alumina, etc. A little of the magnesium carbonate thrown down by the boiling will, moreover, become re-dissolved by the time the water has cooled, and hence go to help in the formation of "permanent hardness."

Although the amount of mineral solids which the water contains generally forms an index to the extent of "hardness,"—the latter increasing pretty much *pari passu* with the former—yet this must not be considered as by any means constantly the case, and some saline waters yielding considerable quantities of "total solids" are very "soft"—as seen notably in the case of some Artesian well waters, in which a considerable quantity of sodium salts exist, and mainly contribute to the softness.

The salts causing temporary hardness furnish a loose deposit in boilers, those causing permanent hardness a hard deposit.

The analysis of waters used for steam purposes should include the estimation of temporary and permanent hardness, the total solids, free acid, and sulphates.

### QUANTITATIVE TEST.

#### **Apparatus required.**—

1. A small glass stoppered bottle (of about 250 c.c. capacity) used for shaking up the water and soap solution.
2. A burette graduated in cubic centimetres and parts of cubic centimetres.
3. A glass beaker, with tripod, wire gauze, and triangle lined with pipe-clay.
4. A measuring flask for 100 c.c.
5. A measuring flask for 250 c.c.
6. Filtering apparatus.

**Chemical reagents.** — 1. A standard solution of potassic soap or of good undried Castile soap, made to such a strength that 1 c.c. will exactly precipitate either 1 milligramme of calcium carbonate, or those other soap-destroying agents in the water to an extent which is equivalent to 1 milligramme of calcium carbonate.

14 grammes of Castile soap are dissolved up in a litre of a mixture of equal volumes of methylated rectified spirit and distilled water; it is then filtered and standardised (and re-standardised from time to time) by means of a standard solution of calcium chloride.

The calcium chloride solution is made by dissolving 0.2 gramme of pure crystallised calcite in dilute hydrochloric acid; when this is completely dissolved, evaporate to dryness on a water bath; then add a little distilled water, and again evaporate to dryness, and repeat this treatment several times

to ensure that all the acid has been driven off. The calcium chloride is then dissolved in a litre of distilled water.

The soap solution must be fortified by adding a little strong solution of soap, or weakened by a mixture of water and rectified spirit in the proportion of three volumes of water to five of spirit, until the soap solution registers hardness equivalent to 20 parts of  $\text{CaCO}_3$  per 100,000.

2. A solution of oxalate of ammonia.

#### THE PROCESS.

The *rationale* of the process is as follows:—The soap employed is a result of the combination of an alkali with one or more of the fatty acids, *i.e.*, oleic, stearic, or palmitic. If potash is the alkali employed the result is “soft soap,” consisting chiefly of oleate of potassium; whereas if it be soda a “hard soap” results, consisting mainly of stearate of soda. Supposing, as in this test, “soft soap” be added to water which contains calcium and magnesium salts in solution, then the fatty acids (oleic mainly) will decompose these salts, and combine with the calcium and magnesium, forming chiefly an insoluble oleate of these bases, *i.e.*, calcic and magnesian oleate; and thus the soap has to be added until there is no longer any calcium and magnesium salts to decompose and combine with, when it remains in solution and creates a lather if the water is well shaken up with it. Hence the more calcium, magnesium, etc., salts there are present, the larger amount of soap solution will be required to decompose them, and in consequence, the longer will the production of a lather be delayed—until in some cases, so much precipitate of these insoluble salts has been thrown down, that their very presence alone will delay the formation of such a lather which is subsequently described as characteristic.

1. 100 c.c. of the water are placed within the “shaking bottle.”

2. The burette is then filled up to the 10 c.c. mark with the soap solution, of which 2 c.c. are run into the bottle—when a cloudy precipitate of insoluble calcic and magnesian stearate, oleate, or palmitate is formed. The bottle is then briskly shaken to see if its contents will show a lather.

3. The solution is afterwards added in cubic centimetres, and the bottle well shaken up after each fresh addition, until a certain definite and characteristic lather is at last produced. The air should be sucked from the bottle (with a glass tube) from time to time, so as to remove any carbonic acid which has been liberated. Sufficient soap solution has been added when the conditions created in the contents of the shaking bottle are the following:—

4. (a) The contents of the bottle on being shaken give only a faint dull soft sound.

(b) After shaking, small particles of the lather cling to, and slowly descend the sides of the bottle.

(c) There is a quarter inch of fine uniform lather, and when the bottle is placed on its side, the lather presents a thin, unbroken surface, after the lapse of five minutes.

5. From the number of cubic centimetres of soap solution required for the 100 c.c. of the water, the amount of calcium carbonate, or its equivalents in soap-destroying power, in the water, is deduced.

6. A deduction of 1 c.c. from the amount of soap solution used must be made in every case, since this amount of soap solution is required to create a similar lather in the same bulk of *distilled* water, which is, of course, free from any of the ingredients which are here considered as furnishing “hardness.”

*Example.*—100 c.c. of water required 15 c.c. of the soap solution to furnish the characteristic lather.

Deduct 1 c.c. which would be required for 100 c.c. of distilled water = 14 c.c.

But 1 c.c. of the soap solution will combine with 1 milligramme of calcium carbonate, or its equivalents.

Therefore 14 c.c. has combined with 14 milligrammes of calcium carbonate, etc.

Therefore the "total hardness" in 100 c.c. of the water is equivalent to 14 milligrammes of calcium carbonate, and 14 milligrammes in 100 c.c. (or 100,000 milligrammes) = 14 parts per 100,000.

*Conclusions to be drawn from the amount estimated.*—The "total hardness" of a water should not exceed 30 parts per 100,000, or it becomes quite unsuitable for washing and cooking purposes; and if it much exceeds 40 it is practically useless in this respect. A soft water may contain up to 10; a hard water from 20 to 30; a very-hard water from 30 to 40 and upwards.

NOTES.—Where the hardness exceeds 20 parts per 100,000, so much precipitate of calcium and magnesium stearate, palmitate, or oleate is created, that it interferes with the formation of a characteristic lather, and leads to an error by over-estimation of the "hardness." In these cases it is necessary to dilute the water with an equal amount of distilled water, *i.e.*, 100 c.c. of distilled water are added to 100 c.c. of the sample, and in the estimation of the hardness 2 c.c. are deducted from the soap solution used up, instead of 1.

The soap solution is unstable, and after a week or so varies in strength, and deposits a thin sediment; to avoid error, therefore, it must always first be standardised when an interval of a week has elapsed since it was last used.

When the results are expressed in "degrees" upon Clark's scale, 1 degree (Clark) simply corresponds in this country to 1 grain of calcium carbonate per gallon, *i.e.*, of 1 part of  $\text{CaOCO}_2$  in 70,000. In France, however, a degree signifies 1 part of  $\text{CaOCO}_2$  in 100,000; and in Germany one part of  $\text{CaO}$  in 100,000.



The "total hardness" having been found, the next step is to estimate the "temporary" and the "permanent hardness."

1. 250 c.c. of the water are measured out and poured into a glass beaker, and boiled for about half an hour, or until about two-thirds of its original bulk remain.

2. After the water has been placed aside, the mouth of the flask closed, and its contents allowed to cool, all the calcium and magnesium carbonate, and most of the iron, would be contained in the precipitate noticeable at the bottom of the beaker—but a little of the magnesium carbonate will have become re-dissolved. It is the supernatant fluid which contains the "permanent hardness," the "temporary hardness" which has been separated being represented by the deposit.

3. From the beaker the cooled water is gently returned to the measuring flask, great care being taken to disturb the precipitate (which is left behind) as little as possible. The water is then made up to its original bulk by filling up to the 250 c.c. mark with recently boiled distilled water, and the loss by evaporation during the boiling is thus made good; or a reflux condenser may be used while the water is boiling.

4. 100 c.c. of this water is then filtered through a fine filter paper and tested for its "hardness" in the same manner as the original water was, and this will now constitute the "permanent hardness."

5. If the "permanent hardness" be then subtracted from the "total," the remainder will represent the hardness separated by the boiling, *i.e.*, the "temporary hardness."

Assuming that the permanent hardness is represented by (7—1) 6 c.c. of the soap solution.

The total hardness was 14 parts per 100,000  $\therefore$  the "temporary hardness" =  $14 - 6 = 8$  parts per 100,000.

*Conclusions to be drawn from the amounts estimated:—*  
The greater the proportion of "temporary" to "permanent" hardness the better, for while the former is remediable by

several efficient processes—and notably Clark's—the latter in addition to being constituted of more objectionable salts from a health standpoint, is extremely difficult to remedy—although such processes as Maignen's and Howatson's effect something in this direction by the addition of caustic soda, which, by decomposing the earthy sulphates, tends to reduce the “permanent hardness.” The “permanent hardness” in a good water should not exceed 10 parts per 100,000, and any excess over this amount renders the water undesirable for drinking purposes.

If it be desired to know the amount of *hardness due to magnesium salts* in a water, where the “total hardness” is known to be due to calcium and magnesium salts and free carbonic acid, it is necessary to first precipitate and remove all the calcium salts in the manner shown in Chapter VIII.

Wanklyn has pointed out that whereas lime reacts immediately upon solution of soap, magnesia requires the lapse of time; and that one equivalent of magnesia consumes as much soap solution as one and a half of lime. If magnesium salts exist in *quantity* in the water, the lather is also modified in other directions, a thin, fine, dirty film forms upon the surface, which soon breaks up, and which cannot be mistaken for the typical pure white lather. In these cases the water must be diluted considerably with distilled water. Bearing in mind, then, the longer time taken for magnesia to react, the presence of this film will warn the operator that he must proceed slowly and shake more thoroughly—the while he adds the soap solution.

*Note.*—In Clark's process lime is added, in quantities depending upon the free carbonic acid in the water, in order that it may combine with this acid which holds the calcium and magnesium carbonates in solution, and the whole be precipitated as insoluble calcium and magnesium carbonates. When the lime is added in too large quantities, some

of it remains in solution in the water in an uncombined state (there being no more carbonic acid to combine with it). Since this is, to an extent, injurious from a health point of view, frequent attempts should be made to discover this state of things in water treated by Clark's process. A ready and simple method of detecting uncombined lime is by adding a few drops of a solution of silver nitrate to some of the water in a test-tube, when if such be present, the cloudiness created, instead of being white and clean (from the silver chloride), becomes dirty and brown (an oxide of silver being formed).

The Rivers Pollution Commissioners in their Sixth Report give the following classification of waters as to their softness:—1. Rain water. 2. Upland surface water. 3. Surface water from cultivated land. 4. River water. 5. Spring water. 6. Deep well water. 7. Shallow well water. And they found that the following formations almost invariably furnish hard waters:—1. Calcareous silurian. 2. Calcareous Devonian. 3. Mountain limestone. 4. Calcareous rocks of the coal measures. 5. New red sandstone. 6. Conglomerate sandstone. 7. Lias. 8. Oolites. 9. Upper greensand. 10. Chalk.

## CHAPTER VII.

## THE POISONOUS METALS.

THOSE poisonous metals for which it is commonly necessary to test a water, are lead, iron, copper and zinc.

Water, under favourable circumstances, will be found most generally to take up these metals either from the pipes through which it has been made to flow, from the receptacles in which it has been stored, or from the materials used in making or repairing the joints of such pipes or cisterns ; but in addition, such metals may gain access from trade processes carried on by river sides, or from metalliferous mines within the district, or in the case of iron commonly from the soil or strata permeated by the water.

**Lead** is taken up from the pipes and cisterns made of this material. The action of the water upon this metal is primarily an oxidising one, a loose coating of oxy-hydrate of lead being formed ; a high degree of oxygenation will therefore favour the plumbo-solvent action of water. The lead oxide is practically insoluble in those waters which do not contain some free acid, but when this is the case (as notably in peaty waters), the lead salt is carried away in solution—in other cases it is removed in suspension. Acidity may also be furnished to waters by excess of carbonic acid, or from the sulphuric acid that results from the oxidation of iron pyrites.

The solubility of the oxy-hydrate of lead is materially affected by the presence of sulphates, chlorides, etc., in water which is not acid, and nitrates favour the oxidation of the metal to oxy-hydrate. Since waters containing carbonates

(of lime, etc.), provide a coating of carbonate of lead to the surface of the metal, which coating is quite insoluble in such waters, it follows that soft waters are the great lead carriers. Soluble phosphates in the water will also protect the metal to a marked degree.

The plumbo-solvent action of water is favourably affected by a rise in temperature up to about 120 deg. F., and by the presence of a large number of micro-organisms.

A chalybeate water generally contains its **iron** in the form of bi-carbonate, *i.e.*, the iron is converted into carbonate, and this is held in solution by free carbonic acid; on prolonged exposure to air, or by applying heat, hydrated ferric oxide, or "rust" is thrown down ( $4\text{FeCO}_3 + \text{O}_2 + 2\text{H}_2\text{O} = 2\text{Fe}_2\text{O}_3\cdot\text{H}_2\text{O} + 4\text{CO}_2$ )—rust being insoluble in water containing no free acid. Upland, moorland, and some other waters (as those from the green sand and new red sandstone), generally contain traces of iron, which are taken up from the soil and strata permeated.

**Copper** is sometimes given to water by culinary utensils made of this metal, for a small amount of copper is dissolved when water which contains common salt, acid (vinegar, etc.), fatty or oily material, is boiled in contact with it. It is an occasional, and highly condemnatory practice, for cooks to place a penny into the saucepan in which vegetables are boiled, in order to improve their colour, and the writer has recorded an instance where this practice gave rise to symptoms of copper poisoning in a household.

**Zinc** is most generally taken up from the galvanised iron cisterns or pipes with which the water has come in contact. It generally exists in water in the form of bicarbonate, but is never present except in very small traces. Galvanised iron must not be held to entail much danger in its use, unless the water contains much free carbonic acid, since under common conditions the zinc oxide or basic carbonate forms and pro-



protects the subjacent metal from further action. Zinc, as sulphate, has been observed in considerable quantity in certain springs in the south of France, New Zealand, and America.

Lead and copper, being cumulative in their actions, small and apparently insignificant traces may in time lead to symptoms of chronic poisoning. The smallest possible trace, therefore, of these metals should theoretically be considered sufficient to render condemnation of the water justifiable. Iron is not dangerous to the same extent, and since it gives indication of its presence when in such amounts as would make its ingestion undesirable, by imparting a distinct taste to the water, its powers for evil on this account are almost nullified—for people will not, as a rule, drink water that is unpleasant to the taste.

Zinc never exists but in very small traces, and since it is not a cumulative poison, the possibility of danger from this metal is very limited.

**Chromium.**—Potassium chromate and chromic acid are both very poisonous. Chromium may possibly get into water which is polluted from dye-works—but it must be extremely rare that this metal ever gains access to drinking water.

**Manganese and Tin.**—If these metals are found in water, their presence would be regarded with great disfavour. Like **arsenic** and **barium**, they very rarely gain access to water.

**Apparatus required :—**

1. Two white porcelain dishes, and glass stirring rods.
2. Test-tubes.
3. A burette graduated in cubic centimetres and tenths of cubic centimetres.

**Chemical reagents :—**

1. A solution of the yellow chromate of potassium.
2. Freshly prepared ammonium sulphide solution.
3. A solution of the ferrocyanide of potassium (which must

not be stale, or it sometimes gives a bluish green colour with hydrochloric acid alone).

4. A solution of potassium ferricyanide.
5. A solution of potassium cyanide.
6. A solution of potassium sulpho-cyanide.
7. Ammonia water.
- 8.\* Dilute hydrochloric acid.

9. Apparatus and reagents for Marsh's test.

10. A standard solution of lead acetate, 1 c.c. = 1 milligramme of lead ; made by dissolving 1.83 grammes of crystallised acetate of lead in a litre of distilled water.

11. A standard solution of copper sulphate, 1 c.c. = 1 milligramme of copper ; made by dissolving 3.944 grammes of sulphate of copper in a litre of distilled water.

12. A standard solution of ferric chloride, 1 c.c. = 1 milligramme of iron ; made by dissolving 1.004 grammes of iron wire in nitro-hydrochloric acid, precipitating with ammonia, washing and redissolving the ferric oxide in a little hydrochloric acid, and then diluting to a litre.

Each of these standard solutions may be diluted with advantage in some cases a hundred-fold ; so that each contains 0.01 milligramme of the metal.

### THE PROCESS.

1. Evaporate a litre of water to about 200 c.c., after acidulating with a drop or two of hydrochloric acid, and let cool. To about 100 c.c. of the concentrated water, placed in a white porcelain dish, apply a little of the ammonium sulphide solution upon a glass rod. By drawing the rod gently through the water, and noticing any discoloured streak in the latter immediately adjacent to the track of the rod, faint quantities of poisonous metals will be more readily detected than by

\* Dilute acid solutions generally contain about 1 part of strong acid to 3 or 4 of distilled water, unless otherwise stated.

allowing a drop of the reagent to fall into the water and then stirring. The reason for this is that the reagent itself imparts colour, and it is therefore advisable to add as little of it as possible to commence with ; otherwise a faint discoloration caused by a metal may be lost in that created by the reagent.

2. Any evidence of a dark colour appearing in the water denotes the presence of lead, iron or copper, and the reagent should be added until the *maximum* amount of darkening has been produced ; it must be borne in mind that iron when in faint traces only imparts a slight dirty green colour to ammonium sulphide at first, but after a while the black precipitate ( $\text{FeS}$ ) separates. If there is any colour present in the *original* water, a comparison must be made with a similar quantity of the water, placed in another porcelain dish, before it is decided whether any additional colour has been furnished by the ammonium sulphide. Where, however, the colour originally present is marked (as by peat, etc.), it may well obscure, even with these precautions, such a slight amount of lead, iron, or copper, which would otherwise make itself manifest. In these cases it would be well, especially where there is any cause whatever for suspicion, to evaporate 100 c.c. of the water to dryness, burn off at a dull red heat the organic matter from the solid residue (and with it the colouring matter will also be destroyed when this is due to peat) and then having dissolved the mineral residue in warm distilled water and hydrochloric acid, to proceed to test the solution.

3. Next divide the darkened water into two parts. To one of these add a drop or two of dilute hydrochloric acid ; if the colour disappears it is due to *iron*, or if it diminishes perceptibly iron is present.

A confirmatory test should then be applied to some of the water in a long, narrow test-tube, *i.e.*, a drop or two of the acid is added, and then a few drops of a solution of the

ferrocyanide of potassium, when the colour of Prussian blue is produced; or another very delicate test is to boil the water with a few drops of nitric acid, cool, and add a little potassium sulpho-cyanide—a blood-red or sherry colour results, due to ferric sulpho-cyanide.

4. If, after adding the hydrochloric acid, the colour does *not* disappear, the metal is either lead or copper. To the other half of the darkened water, add a few drops of a solution of potassium cyanide; the PbS will be unaffected; but the CuS will be completely dissolved.

An excellent confirmatory test for lead is to add to some of the water in a long test-tube, a few drops of the yellow chromate of potassium—when, if lead be present, an opacity appears in the water (due to the formation of lead chromate) and the yellow colour appears more of a canary hue. The reaction is, however, comparatively slight, and difficult of appreciation with faint traces of lead, which will often be missed by this test unless a careful comparison is always instituted with another test-tube, containing a similar amount of pure water and reagent. Under these conditions so little as  $\frac{1}{25}$  grain to the gallon can *sometimes* be detected *in the original water*, if the water is either quite clear at the outset, or has been rendered so by filtration.

The ammonium sulphide reaction is itself an extremely delicate test for lead, and to the novice will not be found to present the same difficulties as the chromate of potassium test; but since copper and iron will furnish the same reaction, their presence must be most carefully excluded before lead is ascribed as the cause of the darkening.

It is important to bear in mind that iron is liable to be present along with lead, or even copper, and may contribute to the darkening created by the ammonium sulphide; the possibility of this taking place may be, however, excluded by adding a drop of dilute hydrochloric acid to the water—for this has been seen to prevent or remove any darkening fur-

nished by an iron salt. Or the iron may be separated by adding nitric acid, evaporating to a small bulk, and precipitating the iron with excess of ammonia and warming; collect the precipitate of ferric oxide, wash, dissolve in nitric acid, reprecipitate with ammonia, filter and wash; boil the filtrate until all the ammonia is driven off, and then test for lead or copper.

Dr. Garrett has devised a most delicate test for lead. The author of the test directs that  $\frac{1}{2}$  ounce of water be placed into a narrow test-tube, and one drop of dilute hydrochloric acid be added to obliterate iron. One drachm of strong, freshly prepared sulphuretted hydrogen water, and  $\frac{1}{2}$  grain of sulphate of barium are then added, when the whole is well shaken and set aside. The insoluble barium sulphate (used in the form of powder) collects, and carries down with it the sulphide of lead, "the presence and quantity of the lead being declared by the degree of colour imparted to the white powder." This white powder in settling forms two layers, and it is in the upper one of these that lead imparts a coloration, varying from "the palest Indian grey or buff to the darkest brown."

5. To confirm the test for copper, a drop or two of a solution of the ferrocyanide of potassium should be added to some of the water after it has been acidulated with a drop of dilute hydrochloric acid (since the reaction does not take place in an alkaline liquid). If copper be present, a bronze coloration and precipitate of cupric ferrocyanide appears, which is best detected by holding the test-tube against a white background.

A further corroborative test consists in adding to a little of the water in a test-tube, a drop or two of ammonia, when a faint blue colour is furnished, due to the formation of a basic salt of ammonia and oxide of copper—which will often be missed unless it be looked for through the depth of the water on to a white background.



6. When no darkness is created in the water, and it is judged desirable to test for zinc, some of the concentrated water should be first tested with a few drops of ammonium sulphide. The white precipitate created in the presence of zinc, when once seen will always be recognised, since it has marked characteristics, *i.e.*, it is of a flocculent, curdled, and gelatinous appearance.

As a confirmatory test, the ferrocyanide of potassium, with excess of dilute hydrochloric acid, will give a white gelatinous precipitate of zinc ferrocyanide, insoluble in dilute acids; and potassium ferricyanide furnishes a rusty yellow precipitate of zinc ferricyanide, soluble in hydrochloric acid and ammonia.

7. The presence of arsenic has very rarely to be tested for, but when it is desirable or necessary to do so—as notably in the case of water taken from the neighbourhood of manufactories of aniline colours, or where the soil is rich in arsenic—Reinsch's test is the most readily performed, but Marsh's test is more delicate.

**Marsh's Test for Arsenic.**—A litre of water is rendered alkaline by solid sodium carbonate (free from arsenic), evaporated nearly to dryness, and then introduced into Marsh's apparatus. The nature of this is indicated in fig. 27; the bulbs at A are intended to receive the water carried over with the current of gas, B is packed with calcium chloride, and serves as a drying medium.

Some pure zinc is placed inside the bottle, together with a little distilled water, and the cork inserted. Some pure sulphuric acid is then poured down the vertical glass funnel, when, as the result of a chemical interchange, hydrogen gas is liberated, and will escape at D. After about a minute, so as to allow time for the complete displacement of the original air in the apparatus, the gas may be lighted at this point, and if the apparatus and reagents are free from arsenic, it will

burn with an almost colourless flame, which will not blacken or brown a piece of clean porcelain when held in it. Having proved the absence of arsenic in the reagents, the concentrated water is poured down the vertical funnel, and if any arsenic be present, it very shortly furnishes arseniuretted hydrogen, which gives the flame a characteristic bluish or lavender tint. If a piece of cold white porcelain be brought against this flame, a steel grey metallic mirror of arsenic is deposited, which is soluble in a solution containing sodium hypochlorite and sodium chloride.

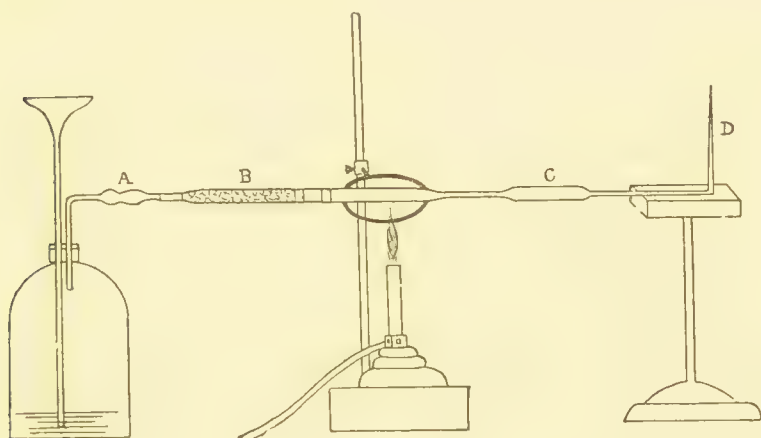


FIG. 27.—Marsh's apparatus.

If the flame is extinguished, and the gas, which will be observed to possess a garlic odour, passed into a solution of nitrate of silver rendered slightly acid with nitric acid, a deposition of silver ensues, and after filtration the solution turns yellow when ammonia is added in drops.

Finally the hard glass tube is heated at about its middle with a spirit lamp, and a brownish-black arsenical deposit ("mirror") occurs in the contracted part just a little behind the heated part of the tube. The mirror varies in lustre and colour according to the amount of arsenic present, and an approximate quantitative estimation may be made by match-

ing the mirror obtained in a certain time with a series of standard mirrors produced from known quantities of arsenic in the same way, and in the same space of time.

Arsenic may be distinguished from antimony by the following tests:—

1. Arseniuretted hydrogen has a strong garlic smell, and burns with a lavender flame; antimoniuiretted hydrogen is inodorous, and burns with a greenish white flame.

2. All arsenical spots are deposited behind the part of the tube heated; antimony spots partly before.

3. Arsenical spots, or mirrors, are brighter than those of antimony.

4. The mirror, or spots when due to arsenic, are completely soluble in a solution containing sodium hypochlorite and sodium chloride.

Marsh's test may be extended by removing with a file a piece of the glass tube which contains the arsenic spots or mirror, breaking this up, and placing inside a long dry test-tube. If the test-tube, held nearly horizontally, with its mouth partially closed by the thumb, is heated below, the arsenic will volatilise, and deposit on the cool part of the tube as arsenious oxide. If this is examined under a magnifying power, clear crystals of perfect or imperfect octahedra and tetrahedra are seen.

**Tin.**—A litre of water should be evaporated to a solid residue, and the tin dissolved out from this by warming with a little strong hydrochloric acid; dilute a little and boil for a long time with metallic copper to make certain that the tin exists in a stannous condition; decant and add excess of a solution of mercuric chloride, when a silky looking cloud of mercurous chloride appears. If a mixture of ferricyanide of potassium and ferric chloride be added to a solution containing stannous oxide or chloride and hydrochloric acid, a precipitate of Prussian blue results from the reduction of

the ferri to ferrocyanide; if no other reducing agents are present, this is very delicate. Sulphuretted hydrogen yields a dark brown precipitate with stannous salts, soluble in potassic hydrate.

**Chromium.**—A good test is to collect the residue from a litre of water, and fuse it with potassium nitrate and sodium carbonate so as to produce the yellow chromate, and this in a neutral solution yields a purple precipitate with excess of silver nitrate. Slight traces may be detected by concentrating the water to a very small bulk, and then letting it drop upon a thin layer of ether floated on a dilute solution of peroxide of hydrogen acidified with sulphuric acid—the blue colour that forms in the lower solution passes over to the ether upon slight agitation.

**Manganese.**—A delicate test (Wanklyn) is to evaporate a litre of water to a small bulk, nearly neutralise with hydrochloric acid, and treat with a few drops of peroxide of hydrogen solution, when a brown precipitate forms in the presence of manganese.

Having thus detected the presence of a poisonous metal, it becomes necessary to estimate the extent to which it exists in the water.

#### THE PROCESS.

1. Measure out "a miniature gallon" (70 c.c.) of the concentrated water which has been found to contain, say, lead, and pour into a white porcelain basin.

2. Place a similar amount of distilled water into several precisely similar basins, to which different amounts (from 0.1 to 1.0 c.c.) of a standard solution of the metal have been added.

3. To each basin add—say two drops of the ammonium sulphide solution, and well stir with a glass rod, each rod being carefully reserved to its own water.

4. Note the amount of the standard solution required to effect a match with the water under examination, and this will represent the amount of lead in the water.

*Example.*—The amount of standard solution required by the 70 c.c. of distilled water to match the coloration in the sample of lead polluted water was 0.2 c.c.

But 1 c.c. = 1 milligramme of lead.

$$\therefore 0.2 \text{ ,,} = \frac{1}{5} \text{ ,,} \text{ ,,}$$

$\therefore$  there is  $\frac{1}{5}$  milligramme of lead in 70 c.c. of the concentrated water.

70 c.c. is "a miniature gallon" in which the milligrammes represent grains in the Imperial measure.

$\therefore$  there is  $\frac{1}{5}$  grain of lead to the gallon of the water.

But the original water was concentrated from a litre to 200 c.c.,  $\therefore$  the  $\frac{1}{5}$  grain of lead represents  $\frac{1}{25}$  of a grain to the gallon in the original water.

Since the method of making a quantitative estimation of lead is so precisely similar to that employed with copper and iron, it will suffice if mention is made of the only and obvious difference, *i.e.*, that of the reagent employed. The standard solution must of course consist of a salt of the metal to be estimated in each case:—For lead, a solution of lead acetate has been used; but for copper, the standard solution is made of copper sulphate; for iron, of ferric chloride.

The quantitative estimation of zinc can be conveniently made by taking a measured quantity of the concentrated water (which is found to be free from other poisonous metals), adding ammonium sulphide solution, and collecting the precipitate of zinc sulphide on a filter; this is then well washed with dilute ammonium sulphide, dried, ignited in a weighed capsule at a bright red heat, allowed to cool, and finally weighed as oxide. Or a very approximate estimation may be made by preparing a standard solution of zinc (4.42 grammes of the sulphate to 1 litre of water; 1 c.c. = 1 milligramme



Zn), and matching in the manner indicated above, the turbidity produced in 100 c.c. of the concentrated water to which a small measured quantity of potassium ferrocyanide has been added.

*Conclusions to be drawn from the amounts estimated.*—Opinion has been a good deal divided as to the amounts of the poisonous metals in a water which may be considered as dangerous. In the case of lead  $\frac{1}{10}$  grain to the gallon is generally accepted, and  $\frac{1}{20}$  is advocated by some excellent authorities. It would appear best—because it is safer—rather to make an over-estimation of the danger than to run the risk of erring in the other direction, and in the case of the poisonous metals which are cumulative in their action, it is difficult to see what amount, however small, can be accepted as harmless.

There is evidence that the system rapidly becomes habituated to copper salts, but this metal should not exceed  $\frac{1}{10}$  grain to the gallon in drinking waters.

With regard to zinc, which is not a cumulative poison, a little more latitude may be allowed; but the faintest traces of arsenic—an exceptionally poisonous and somewhat cumulative metal—should suffice to condemn the water.

Iron should under no circumstances exceed  $\frac{1}{4}$  grain per gallon, and that is too high to be desirable except when the water is taken solely for medicinal purposes—as this quantity will provoke dyspepsia, general malaise, headache, etc., in most people, after a time.

#### NOTES UPON THE PROCESS.

Though the metals lead, copper, and iron, when existing in very faint traces, will generally be detected by testing the original water, yet in order to be certain it is generally desirable to reduce the bulk of the water—and thus concentrate their solutions as it were—by evaporation in a large porce-

lain dish. In the case of zinc it will *always* be necessary to considerably reduce the original bulk of the water.

A litre of water may be evaporated to 100 c.c. by previously marking a narrow beaker at the precise level to which 100 c.c. of water reaches, and boiling the litre of water until it is reduced to this level.

The quantitative estimation of iron may also be made from the ferrocyanide of potassium reaction, and that of lead from the chromate of potassium reaction; but, as a rule, lead, copper, and iron are more closely estimated from the colour furnished by their respective sulphides. Only just sufficient of the reagent should be added to create the maximum amount of colour, for a large excess will give a yellowish tinge to the water, and this impairs very materially the facility of effecting a colour comparison. In every case of performing such colorimetric tests it must not be forgotten that it is essential to add similar amounts of reagents to both the water and the comparison solution.

Iron may be estimated gravimetrically in the following manner:—

The residue of 500 c.c. of the water is treated with strong hydrochloric acid, and well washed with boiling distilled water; then add two drops of nitric acid to the filtrate, boil, and add ammonium chloride solution and a slight excess of ammonia; let the precipitate settle; pour the supernatant liquid through a small filter, redissolve the precipitate in the least possible quantity of hydrochloric acid, and again add ammonia. Transfer the precipitate to the filter, wash with hot water, dry, ignite and weigh as sesquioxide ( $\text{Fe}_2\text{O}_3$ ), and this  $\times 0.7 = \text{Fe}$ .

## CHAPTER VIII.

## THE NON-POISONOUS METALS. SULPHATES. PHOSPHATES.

*The Non-Poisonous Metals.*

FOR the ordinary purposes of a hygienic analysis, nothing of any practical value is gained by prosecuting an investigation under this head beyond what is necessary to determine the presence and amount of calcium, magnesium, and silica.

The presence of **Calcium** salts, which mainly exist as the bicarbonate and sulphate in a water, may be discovered by creating the white precipitate of the oxalate of calcium, by adding a solution of oxalate of ammonia, followed by ammonium chloride and sufficient ammonia to furnish a slight ammoniacal odour. The ammonium chloride serves to hold any magnesium present in solution. If it be desired to make a quantitative estimation, a measured quantity of water, say 250 c.c. (concentrated from a litre), must be thus treated and set aside in a warm place for several hours, and the precipitate carefully filtered—until the filtrate is quite clear—through a *Swedish* filter paper (of which the weight of ash yielded by ignition is known). The oxalate of calcium precipitate remaining on this filter paper is thoroughly washed with hot distilled water, and afterwards dried in the hot-air oven at a temperature of 220 deg. F. The filter paper should then be folded up, placed in a small porcelain crucible (previously weighed), covered by a lid, and then ignited to dull redness—at first gently so as to obviate spurting and loss; the lid should be removed after a little so as to permit free access of air. When the filter paper has been entirely destroyed

("burnt off"), and the capsule and its contents allowed to cool under a desiccator, the weight is taken. The weight found, minus that of the crucible and the ash of the filter paper, represents the weight of the calcium carbonate present—to which form the oxalate has been reduced by ignition.

**Magnesium Salts** mainly exist in water in the form of the carbonate and sulphate, and chiefly in that collected from the dolomite strata; if these salts exceed 5 grains per gallon they are apt to cause dyspepsia and diarrhoea in those unaccustomed to the use of such waters. The presence of magnesium salts may be best ascertained by precipitating all the lime present in the water, by means of a solution of oxalate of ammonia, ammonium chloride, and ammonia, as before, and filtering until the filtrate is perfectly clear and free from calcium. The filtrate, slightly acidified with hydrochloric acid, should next be concentrated by boiling, and then a few drops of a saturated solution of phosphate of sodium are added, and sufficient ammonia to create strong alkalinity. The whole is well stirred up with a glass rod, and then set aside for several hours, when a crystalline precipitate of a double phosphate of magnesia and ammonia (ammonium-magnesium phosphate or "triple phosphate") is formed. This may be collected on a filter, washed with dilute ammonia, dried, ignited at a red heat, and weighed when cold as pyrophosphate ( $\text{Mg}_2 \text{P}_2 \text{O}_7$ ), to which the red heat reduces the double salt; this  $\times 0.2155 = \text{Mg}$ .

In those cases where the magnesium salts are present only in minute traces, no definite precipitate results, but the points where the stirring rod has touched the glass appear as white streaks, readily soluble in hydrochloric acid.

The *amount* present can also be very approximately estimated from the hardness which it will create when a water, perfectly freed from calcium, is tested by the soap solution

(*vide* "Hardness"). Supposing, for instance, 5 c.c. of the soap solution are required to satisfy the hardness remaining in 100 c.c. of the water : deduct 1 c.c. (the amount of solution required for an equal bulk of pure distilled water) = 4 c.c.

But 1 c.c. of soap solution = 1 milligramme of calcium carbonate.

∴ 4 c.c. = 4 milligrammes of calcium carbonate.

∴ The hardness due to magnesium salts in 100 c.c. of water is equivalent to 4 milligrammes of calcium carbonate. But 1 part of calcium carbonate is equivalent to 0.56 part of magnesium carbonate (Wanklyn).

∴ the magnesium would be represented by  $4 \times 0.56 = 2.24$  parts of magnesium carbonate in 100,000 of water, or  $\frac{7 \times 2.24}{10} = 1.5$  grains per gallon.

Magnesium carbonate has been estimated as high as 9 grains per gallon by Wanklyn and Playfair in Sunderland water.

The estimation of **silica** may become of importance, having in view the fact that, as Dr. Tidy pointed out, its presence diminishes the plumbo-solvent action of water. In quantities it may also create gastro-intestinal irritation and diarrhœa.

It generally exists in the form of insoluble silicate of alumina (clay), or of the soluble alkaline silicates.

A measured quantity of water, say 500 c.c., is acidulated with hydrochloric acid, and then evaporated to a solid residue ; this is treated with strong hydrochloric acid, and then well washed with boiling distilled water, when the residue collected on a filter is dried, ignited, and again treated with the acid and washed as before ; any residue ultimately left will consist of most, if not all, of the silica originally present in the water, and the white gritty powder of silica ( $\text{SiO}_2$ ) may be dried, ignited, and weighed.



## SULPHATES.

Sulphates exist in most waters, especially selenitic\* ones, and are either derived from the strata over or through which the water has passed, or from the sulphur contained in organic pollution (urine, etc.). The rain water collected in large towns yields small amounts derived from the sulphurous acid in the smoky atmosphere. Sulphates sometimes result from the oxidation of metallic sulphides (chiefly iron pyrites), which exist as such in certain strata. They mainly exist as calcium and magnesium sulphates, and less commonly as sodium sulphate, and any of these, when present in large amount, tend to cause diarrhœa and dyspepsia in those unaccustomed to their use.

Waters collected from the limestone and dolomite formations always contain a marked quantity of sulphates, but not so the water from the chalk. The sulphates in limestone may reach to 20 parts per 100,000, and consist mainly of calcium sulphate; while those of the dolomite consist to a less extent of calcium sulphate, and partly of magnesium sulphate.

The so-called "sewage-fungus" ("*Beggiatoa alba*") is found where sulphates abound, and as the action of the fungus is to reduce these to sulphuretted hydrogen, there is a consequent production, to a greater or less degree, of the characteristic and offensive odour of this gas.

**The Test.**—A few drops of chloride of barium solution and of dilute hydrochloric acid are added to the water in a long test-tube, which is then left to stand for a few minutes, when an opacity or precipitate of the sulphate of baryta is created with even very small quantities of sulphates.

If a *quantitative estimation* is desired, 100 c.c. of water (concentrated from a litre) should be strongly acidified with

\* Selenite is a natural foliated or crystallised sulphate of lime.

hydrochloric acid, heated to boiling, and an excess of a hot solution of barium chloride cautiously added with constant stirring, until the maximum turbidity is furnished. The precipitate formed is collected on a small Swedish filter paper, washed, ignited at a moderate red heat, and weighed as barium sulphate, the washing of the precipitate being continued until the filtrate no longer gives a turbidity with silver nitrate.

To express the result in terms of sulphuric acid ( $\text{SO}_3$ ) it is necessary to know that every part of barium sulphate represents 0.3436  $\text{SO}_3$ . In drinking waters the amount of  $\text{SO}_3$  should be under 10 parts per 100,000.

#### PHOSPHATES.

The phosphates found in water are commonly those of potassium, sodium, and ammonia, and their double salts. The presence of these affords corroborative evidence of animal contamination; but they may only exist in small amounts in those waters generally used for drinking purposes, even when these be dangerously polluted, for phosphoric acid is eagerly retained by soils that the water may have come in contact with. When this point is considered in conjunction with the facts that many waters contain faint traces of phosphates, that these may also have their origin in strata—chiefly sandstone—permeated, and that they have also been found to be present in some marshy waters, it is difficult in some cases to conclude as to what interpretation shall be placed upon their presence when in traces. When in marked amount they may be taken as a certain sign of organic pollution; their complete absence, on the other hand, is no guarantee of a water's freedom from such pollution. More than 0.05 parts of  $\text{P}_2\text{O}_5$  per 100,000 would always be regarded with suspicion.

There is, moreover, another important consideration connected with their presence, *i.e.*, that they form food-pabulum for bacteria and fungi, and they must always indicate possibilities of danger on this account.

In every case before a test is applied for phosphoric acid, the water should be reduced from a large bulk to a very small one by evaporation, and it is even preferable to dissolve it out from the ash by nitric acid.

**The Test.**—100 c.c. of water are acidified with a little nitric acid and evaporated to a solid residue; the mineral ash is next obtained at as low a temperature as possible. The phosphoric acid is dissolved out from this ash with about 3 c.c. of warm distilled water, to which a few drops of dilute nitric acid have been added; filter through a paper previously washed with dilute nitric acid, mix the filtrate with 3 c.c. of molybdic solution, gently warm and set aside for 15 minutes at a temperature of about 80 deg. F. If “traces” of phosphates are present a faint greenish yellow turbidity will be noted; if “heavy traces,” a marked yellow precipitate falls. If the precipitate, which consists of yellow phospho-molybdate, is appreciable, it may be collected, washed with distilled water, dissolved in ammonia, and precipitated with magnesia mixture. This precipitate is then collected and washed with 2½ per cent. ammonia, ignited, and weighed as  $\text{Mg}_2\text{P}_2\text{O}_7$  (magnesium pyrophosphate).  $\text{Mg}_2\text{P}_2\text{O}_7 \times 0.64 = \text{P}_2\text{O}_5$ .

### *Solutions required.*

**Molybdic solution.**—Dissolve 1 part of pure molybdic acid in 4 parts of  $\text{NH}_3$  (S.G. 0.960); filter, and pour with constant stirring into 15 parts of nitric acid (S.G. 1.2); let stand in the dark for a few days; carefully decant, and keep in the dark.

*Magnesia mixture.*—55 grammes of crystallised magnesium chloride are added to 70 grammes of ammonium chloride, and the whole dissolved in 1 litre of 2½ per cent. ammonia. About 15 c.c. of the mixture should be used to precipitate 0.1 gramme  $P_2O_5$ .

## CHAPTER IX.

## THE SOLID RESIDUE.

By this term is implied the substances which are held in solution in the water, and which, when the latter is evaporated, remain behind as a solid residue.

The mineral salts which a water may contain are taken up in its course through or over the soil or strata it comes in contact with (with the exception of traces of sodium chloride which may be taken up from the atmosphere).

In regard to the principles which guide chemists in the hypothetical association of the acids and bases which form these salts, it is assumed that their combinations are governed by their respective affinities; that is the strongest acid is assumed to be combined with the strongest base, due attention being also paid, however, to the greater or less degree of solubility of the salts—since it is well known that this exercises a considerable influence on the manifestations of the force of affinity.

Thus it is assumed that the chlorine is combined with sodium; any excess (which is rare) is allotted to potassium, or in the absence of potassium to calcium. If there is excess of sodium it is assumed to be in combination with sulphuric acid, any excess of which is allotted to calcium and magnesium; and calcium and magnesium, if not combined with sulphuric acid, nitric acid, or chlorine, are in the form of bicarbonates.

Nitric acid is held to be combined with ammonia, and when there is excess it is allotted to either soda or lime or magnesia,



as circumstances indicate—in waters which are found to be comparatively free from organic matter.

The other constituents of the mineral residue, being in such small amounts, are generally not grouped as salts, the silica, iron, alumina, nitrous acid, and phosphoric acid being returned as  $\text{SiO}_2$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{N}_2\text{O}_3$ , and  $\text{P}_2\text{O}_5$ , respectively.

To give a simple example :—

A mineral water is analysed and found to contain :—

	parts per 100,000
Sulphuric acid ... ..	186.07
Soda ... ..	66.72
Magnesia ... ..	52.76
Chlorine ... ..	13.40
Lime ... ..	6.68
Carbonic acid ... ..	2.12
	<hr/>
	327.75
	<hr/>

These constituents would be expressed in combination as follows :—

Sulphate of magnesium ... ..	158.28
Sulphate of sodium ... ..	132.86
Sulphate of calcium ... ..	9.69
Chloride of sodium ... ..	22.11
Carbonate of calcium ... ..	4.81
	<hr/>
	327.75
	<hr/>

The estimation of the amount of residue is not generally of great practical value, from a hygienic point of view, though often when we come to incinerate, valuable corroborative evidence of organic pollution is obtained.

A high amount of mineral solids, consisting as it frequently does in this country largely of calcium carbonate (even up to

50 or 60 parts per 100,000), is not considered necessarily injurious; but if a goodly proportion of the mineral residue is found to be contributed by the salts which form permanent hardness, one is disposed, since these salts are productive of digestive disturbances in many people, to look with disfavour upon the water. Certainly no drinking water should contain more than 10 parts per 100,000 of the sulphates of the alkalies and of magnesium.

Another important question which a large amount of total solids would give rise to, is that of the suitability of the water for washing and cooking purposes, on account of a probable excess of hardness.

The total solids have been estimated higher than even 300 parts per 100,000 in deep well water from the chalk, and sea water contains over 3000 parts per 100,000.

**Apparatus required :—**

1. A copper water-bath.
2. A water oven.
3. A platinum dish capable of holding a little over 100 c.c. of water (say 125 c.c.).
4. A desiccator.
5. Chemical balances.
6. Platinum pointed crucible tongs.

**THE PROCESS.**

If it is required to estimate both the dissolved and suspended solids, the sample of water should be shaken up; but if the dissolved alone are required, the suspended matters must of necessity be allowed to subside, or be separated by filtration through a clean porcelain filter or through about a dozen large purified filter papers, ribbed, and packed rather loosely into a large funnel. As generally performed, it is the total solids *in solution* which are assumed to have been esti-

mated, and such a signification must be attached to the expression "total solids" throughout Part I. of this book.

1. 100 c.c. of the clear water are measured out and emptied into the platinum dish, which is then placed upon the water-bath and covered by a large piece of filtering paper, or a glass bell-jar suspended by means of a retort-holder; the jar is fixed slightly aslant, so that the condensed vapour will all run off at one point, where it can be collected in a beaker. Provision is best made for the escape of steam when the small ringed water-bath is employed (fig. 18), by making a small dent up the side of the platinum dish, so that it shall not fit accurately at this point to the mouth of the water-bath.

2. When the water is evaporated to apparent dryness, the dish is removed, and placed for a few minutes in the hot-air bath (fig. 21), in order that "the solid residue" may be finally dried at 220 deg. F., the object being to remove all adventitious moisture, but not the water which is an essential constituent of the substance as the "water of crystallisation."

To save time, it is more convenient to evaporate the 100 c.c. to about 10 c.c. over a low gas flame, without boiling, and *then* transfer to the water-bath, otherwise the process takes over two hours.

3. The dish is removed from the oven, and then allowed to cool under a desiccator.

4. In a few minutes the dish and its contents are weighed, and the difference between the weight found and that of the clean and empty dish represents "the total solids" in 100 c.c. of water.

5. By means of a pair of platinum pointed crucible tongs the dish is next held in the flame of the Bunsen burner and *slowly* heated to dull redness, when any organic matter will give evidence of its presence by charring in little specks, or, if small in amount, by causing an evanescent discoloured wave to spread over the residue. If large quantities are present,

this organic matter, as it is being burnt off, shows blackened patches which slowly disappear, and gives off dark fumes possessing an odour of burnt hair or horn when nitrogenous animal matter is present, or of burning sugar when the material is vegetable; or a sulphurous acid smell may rarely be detected. When a marked amount of oxidised compounds of nitrogen exist, they often give rise to an evolution of red fumes of nitrogen dioxide. Marked scintillation is sometimes also perceptible—that is to say tiny sparks are emitted. Eventually nothing remains but clear *white* mineral ash, except where iron or manganese is present, the former imparting a reddish tint and the latter a green.

6. The dish is allowed once more to cool under the desiccator, and is then reweighed, and the excess of weight over that of the clean and empty platinum dish consists of mineral ash, and represents “the non-volatile solids.”

7. The weight of “non-volatile solids” deducted from that of the “total solids” represents “the volatile solids.”

*Example.*—The clean platinum dish weighs 44.225 grammes.

The dish + the total solids weigh 44.245 grammes.

$\therefore 44.245 - 44.225 = 0.020$  gramme = the total solids in 100 c.c. of water.

But 100 c.c. of water = 100 grammes.

$\therefore$  there is 0.020 gramme in 100 grammes.

Or  $\times 1000 = 20$  parts per 100,000 of total solids.

After ignition, the dish + contents weigh 44.240 grammes.

$\therefore 44.240 - 44.225 = 0.015$  gramme = “the non-volatile solids.”

$\therefore$  There is 0.015 gramme in 100.

Or  $\times 1000 = 15$  parts per 100,000 of “non-volatile solids.”

The total solids = 20 parts and the non-volatile solids = 15.

$\therefore 20 - 15 = 5$  parts per 100,000 of "*volatile solids*."

For public health purposes it must be extremely rare, in view of the information which is acquired in other steps of the analysis, that a complete analysis of the ash is either necessary or desirable; so that its introduction here would be going beyond the important issues of the subject. It may be pointed out, however, that a few drops of dilute hydrochloric acid will, by creating little or much effervescence, roughly detect the proportion of carbonates present, and will generally dissolve out everything but silica, and the sulphates of calcium and magnesium; that boiling distilled water will mostly dissolve from the residue, the salts of the alkalis and of magnesia, and traces of lime; and that the insoluble residue will consist generally in the main of calcium carbonate, but it also may contain silica, alumina, sulphates of the alkaline earths, phosphate of calcium, peroxide of iron, etc.

In the case of mineral medicinal waters, and those used for commercial chemical purposes, a detailed and complete analysis is requisite; the matter is lengthy and complicated, and reference must be made to a work dealing with quantitative chemical analysis.

If it is required to make a close estimate of "the mineral solids," care must be taken to remove the dish from the flame *immediately* any evidence of organic matter has disappeared, and not to conduct the incineration at a higher temperature than is found absolutely necessary—or there is a considerable loss in the mineral residue by destruction, dissipation, and volatilisation.

Of what may "the volatile solids" consist? *i.e.*, to what may be due the loss by ignition when a strong red heat is applied? Such loss is most generally from the following



ingredients:—Destructible organic matter, ammonia salts (by volatilisation), nitrates and nitrites (lose  $O_2$ ), certain chlorides, such as sodium and potassium chlorides (by volatilisation), combined carbonic acid, and the water of hydrated salts (such as calcium sulphate), which thereby become anhydrous; calcium and magnesium carbonate and magnesium chloride are also decomposed.

A platinum dish is cleansed after use with a little dilute hydrochloric acid, then well washed in pure water, and finally heated to redness in the Bunsen flame. It should be allowed to cool upon a clean porcelain slab.

While igniting a solid residue, care must be taken to guard against loss by spurting or decrepitation. When this is likely to ensue, the capsule must be more gradually heated, and kept at first carefully covered by a lid. In every case the residue must be thoroughly dry before ignition is commenced; it is also desirable to keep the platinum dish on the slant during ignition since this favours air draught.

If the operator has not a platinum dish of 100 c.c. capacity, the best plan is to measure out 100 c.c. of the water, and to keep transferring this, little by little, to the dish, as evaporation proceeds. A vessel must never be weighed while hot, for then it weighs less than its proper weight; this is chiefly due to the circumstance that cool bodies condense a varying amount of air and moisture upon their surface.

The ignited residue should be retained for the estimation of phosphoric acid. A loose white light residue indicates the presence of magnesium.

If it is desired to make an estimation of the suspended matter, a measured volume of the water (a litre) should be passed through a thickness of a dozen large purified filter papers, pleated and packed rather loosely into a large funnel; the suspended matter thus collected is then well washed with distilled water and dried at about 240 deg. F., until a con-

stant weight is obtained. This weight, less that of a dozen similar dried filter papers, is the weight of suspended matter in the litre of water, and results may be checked by estimating the total solids in an aliquot part of the water (previously well shaken) both before and after filtration; the difference will indicate the amount of suspended matter.

The volatile and non-volatile suspended matter may be estimated after incineration, as already shown.

## CHAPTER X.

## ORGANIC MATTER IN WATER.

It has been pointed out that the hygienic analysis of water essentially aims at attaining two objects, viz., the determination of the extent of organic pollution, and the detection of the presence of poisonous metals. The former is the more important and urgent of the two, since the evil consequences following in the wake of organic pollution are generally more suddenly dangerous and more far-reaching than those following upon metallic contamination. By organic pollution is included the fouling of water by both *animal* and *vegetable* material, together with the products of their decomposition; and since the relative significance and danger of these forms of organic matter differ very materially (animal contamination being indicative of far more danger and harm than vegetable), it is important, both by a knowledge of the water's source and by chemical analysis, to discover which form of organic matter is fouling the water, or to what extent they are respectively doing so.

Organic matter gains access to water by manifold channels, many of which will readily occur to the reader, and all of which may be learnt by consulting the best works upon Hygiene and Public Health.

This organic matter, as is well known, has a strong natural tendency to resolve itself, under suitable conditions of temperature, air and moisture, into simpler parts, by fermentation, *eremacausis* and putrefaction. In the process of fermentation numerous minute forms of vegetable life are

developed, which, if they do not start the process themselves, play an important part in fostering and continuing it.

By *eremacausis* is implied the breaking up of organic matter by slow oxidation; in other words the natural "burning-off" of organic matter—its carbon appearing as carbonic acid, its hydrogen as water, and its nitrogen as ammonia, nitric and nitrous acids.

Whereas in these two processes no offensive odour is created, when putrefaction sets in odorous gases are evolved which mostly consist of compounds of sulphur and phosphorus, and—as in fermentation—minute organisms develop, which, in this case, mainly take the form of bacteria, vegetable fungi and the animal infusoria.

The great necessity for a closely proximate estimation of this danger-carrying and danger-breeding material in a water has been recognised for many years, and the difficulty encountered in performing it is both interestingly and instructively exemplified in a perusal of the successive methods to this end which have been advanced and adopted; but the subject must be treated very summarily here.

One method, which was for a long time a popular one, took advantage of the destructibility (and loss) of organic matter by heat. A large bulk of water was evaporated to a solid residue at a low temperature, and this was then heated at a high temperature ("ignited") until all organic matter was burnt off, and nothing but mineral ash remained. The difference in weight of the residue before and after ignition was held to represent the organic matter; until successive chemists pointed out that the loss by ignition included, in addition to organic matter, some of the mineral constituents as well. The organic matter, therefore, was considerably over-estimated by this method, but it had become a popular favourite, and lived, strange to say, for a long time after the

facts which belied its accuracy were generally known and appreciated among chemists.

The effort to estimate the organic matter from the amount of oxygen of which it will deprive the permanganate of potassium (Condy's fluid), was practised almost universally for another long period, and it remains as an auxiliary test for organic matter to this day. The facts, however, that potassium permanganate in solution is so very unstable, that other substances in the water—apart from organic matter—are capable of reducing it, that it will part with its oxygen more readily to the least dangerous (*i.e.*, vegetable) than to the more dangerous (*i.e.*, animal) pollution, and that the oxidisable organic matter bears an unknown and inconstant ratio to the *total* organic matter, have all conduced to some dissatisfaction and mistrust of the test, with the result that further endeavours have been made to find another one more inclusive and reliable in its estimation.

Dr. Frankland has devised a beautiful and ingenious process to meet the want, but it is quite unsuited to the bulk of Health Officers, and there is scope for some error to creep in even with practised hands. He evaporates a measured volume of water to a solid residue, and this is collected in a hard glass combustion tube, mixed with oxide of copper, and burnt in a furnace. The oxide of copper parts with its oxygen to the organic matter, which is completely destroyed, and the carbonic acid, nitric oxide, and nitrogen which result are collected, measured, and returned in terms of "organic carbon" and "organic nitrogen."

A method superior in its facility of execution, against which no important chemical defects can be raised and sustained, and one which has rapidly become popular and accepted among the bulk of chemists, is that known as "The Wanklyn, Chapman and Hall Process." By it an endeavour is made, after computing the amount of "saline" ammonia *originally*



*present* in the water, to estimate the amount of nitrogenous organic matter from the amount of ammonia which can be derived from the breaking up of such matter by boiling with a solution of strongly alkaline permanganate of potassium. No better clue to the presence of organic matter can well be imagined than an estimation based upon the nitrogen produced by its decomposition; but the question which will naturally arise is as to whether *all* injurious organic matter is nitrogenous, and there is no doubt that broadly, in so far as water pollution is concerned, it appears to be so. The composition of many forms of organic matter is so varied and complex, and defies analysis so successfully, that much has yet to be learnt of its chemical constitution.

Of course great consideration is also had in the process to the amount of the "saline" ammonia, and it is held to have *its* origin mainly in recent organic pollution, though it does not, like the "organic" ammonia, necessarily indicate such pollution *actually present as organic matter*, but rather a product of the decomposition of such matter.

While, therefore, the ammonia which is created by the process must always be considered the greatest index of danger—since it is derived from organic matter *actually present* at the time of analysis—the amount of "saline" ammonia is considered of great value as a further index, for most of it, and very nearly *all* in many cases, must be derived from extremely recent organic pollution, although very little of the original organic matter itself whose decomposition furnished it may be present in the water.

It is obvious that no chemical process can determine as to whether the organic matter is living or dead, or whether in the former case it is harmful or not. When it is considered how minute the germs of disease are, it will be seen at once that considerable quantities cannot by themselves materially affect the amount of, say, "organic ammonia"; but since they

would always be associated with organic food-pabulum, the evidence of organic pollution which this would furnish by the analysis sounds the warning note.

Certainly no good water can ever be condemned by this process, nor could a really bad one ever escape condemnation.

The tests for organic matter, in addition to those which aim at detecting it when actually present, also include others whose object it is to detect the *products of organic decomposition*, *i.e.*, oxidised nitrogen in the form of nitrates and nitrites, and those other chemical constituents which, by entering into the composition of organic bodies, gain access to the water along with them, *i.e.*, phosphates, sulphates, and chlorides.

## CHAPTER XI.

## WANKLYN'S PROCESS.

THE principle upon which this process is conducted is that known as a "colorimetric analysis"—a variety of volumetric analysis, in which a reagent, having the power of combining with the substance sought after, and creating a colour with it, is added to a measured quantity of the solution under analysis. The amount of the substance present and thus combined, is then estimated by adding to a similar volume of distilled water and reagent, a solution which contains known quantities of the substance, and this is continuously added until the same degree of coloration is reached. The amount of the substance contained in the quantity of the standard solution used, is, of course, the same as that existing in the solution under analysis.

**Apparatus required :—**

1. A condensing apparatus.
2. A boiling-flask or retort. This must be supported (as shown in fig. 28) upon a four-footed iron stand, across the top of which a piece of wire gauze and a pipe-clay triangle has been placed ; it is then attached to the condensing apparatus by means of a cork which is perforated by the bent end of the smaller tube—the extremity of which is allowed to project about half an inch below the cork into the neck of the bottle.
3. Six Nessler glasses. One of these is represented in

fig. 28 as catching the condensed vapour. These glasses are each marked off at a point which indicates the level to

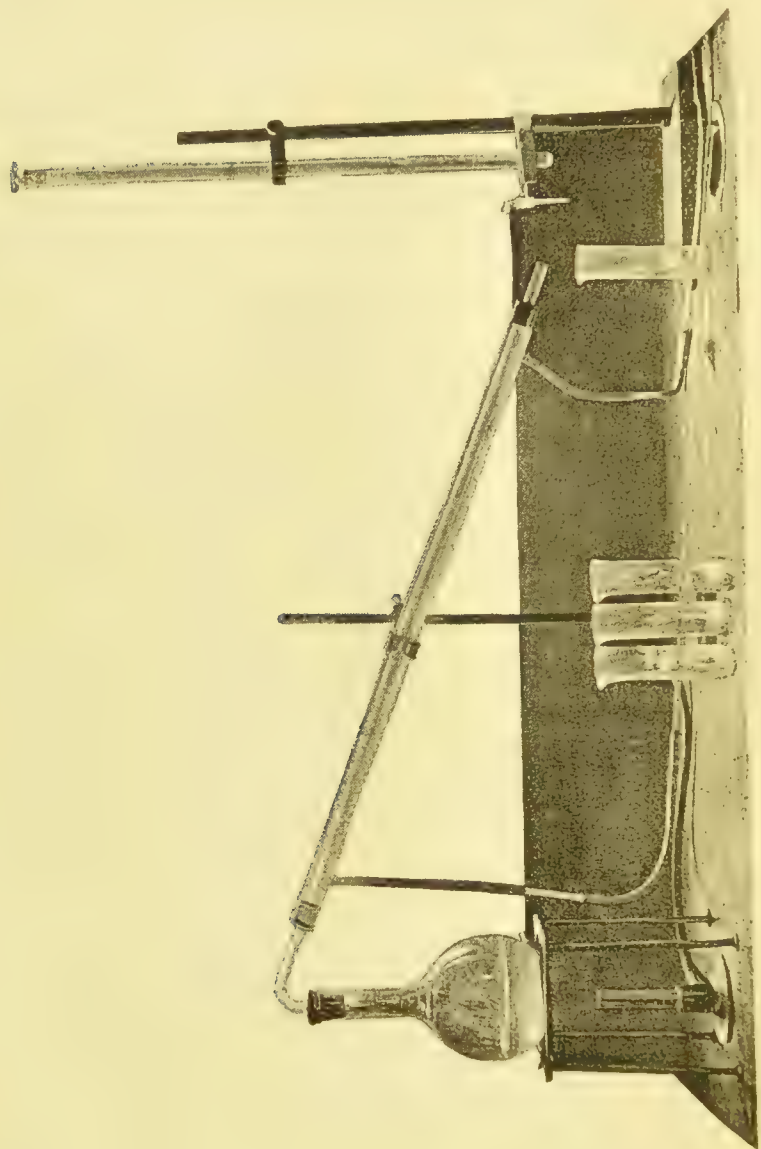


FIG. 28.—The apparatus employed in the estimation of the nitrogenous organic matter by Wanklyn's method.

which 50 c.c. of water will stand in them; they should be made of thin colourless glass, and of precisely similar diameter.

4. A white porcelain slab about six inches square, which is used to facilitate colour comparison.
5. A burette graduated in cubic centimetres and tenths of cubic centimetres, and fixed upon a stand.
6. A glass measuring flask for 500 cubic centimetres of water.

### Chemical reagents required —

1. A standard solution of chloride of ammonium, made to the strength that 1 c.c. contains 0.01 milligramme of ammonia.

The solution is made by dissolving 3.139 grammes of pure chloride of ammonium in a litre of distilled ammonia-free water; if some of this is diluted a hundred-fold with distilled ammonia-free water, it is of the required strength.

2. Nessler's reagent. This consists of a solution of the periodide of mercury in distilled ammonia-free water, the whole being rendered strongly alkaline with caustic potash. When this reagent is applied to a solution containing ammonia, it imparts a colour varying from a faint yellow to a dark brown or amber, and sometimes even a precipitate, according to the amount of ammonia present; and this reaction, which is due to the formation of ammonio-mercuric iodide, may be considered as quite characteristic of ammonia, since it is not shared by any other substance with which we are familiar. It is important, however, to clearly appreciate the fact that it does not react to *organic matter as such*.

The solution of the reagent should have an extremely faint yellow colour, which indicates that it is saturated with the periodide of mercury, and is therefore "sensitive"; should it be colourless and non-sensitive, this can be corrected by the addition of a drop or two of a saturated solution of corrosive sublimate.



Any precipitate of mercuric iodide which settles should not be disturbed when the reagent is being used.

Nessler's reagent is made by "taking 35 grammes of the iodide of potash, 13 grammes of corrosive sublimate, and about 800 c.c. of water. These materials are then heated to boiling, and stirred up until the salts dissolve. That having been accomplished, a cold saturated solution of corrosive sublimate in water is cautiously added, until the red periodide of mercury, which is produced as each drop of the solution falls into the liquid, just begins to be permanent. In this manner we obtain the solution of the iodide of potassium saturated with mercury periodide, and it remains to render it sufficiently alkaline and to render it sensitive. This is accomplished by adding 160 grammes of solid caustic potash, or 120 grammes of caustic soda to the liquid, which is afterwards to be diluted with water, so that the whole volume of the solution may comprise one litre. In order to render the Nessler reagent 'sensitive,' it is mixed finally with a little more cold saturated solution of corrosive sublimate, and allowed to settle" (*Water Analysis*, Wanklyn).

The reagent should be kept in a tight-fitting glass-stoppered store bottle, and small quantities emptied out into a smaller one for use from time to time.

3. 100 c.c. of a strongly alkaline solution of the permanganate of potassium, boiled for a few minutes (ten) on each occasion prior to use, in order to get rid of any traces of ammonia.

The amounts recommended to be used in making up the alkaline permanganate of potash stock solution are:—

Caustic potash, 200 grammes.

Permanganate of potash, 8 grammes.

4. Ammonia-free distilled water, 1 litre + 100 c.c. The solution is boiled rapidly till the 100 c.c. are driven off and the litre remains.

Ammonia-free distilled water is made from the tap water by collecting the distillate from a condensing apparatus (Graham's or Liebig's), after discarding the first quantities which contain any free ammonia (as shown by Nessler's reagent); and care must also be had not to allow the water to boil down too low, or the last amount of distillate may also contain ammonia. For most tap waters, if a litre of such be placed in a boiling-flask, it will only be necessary to discard the first 200 c.c. of distillate, and to stop the process of distillation when about the same amount remains behind in the flask; the rest collected will be ammonia-free, as shown by negative results when a little is tested by Nessler's reagent. Or the ammonia may be first "fixed" by the addition of a very little sulphuric acid, and the distillate from the bulk of the water be collected as "ammonia-free."

#### THE PROCESS.

*Firstly the amount of "free and saline ammonia" is calculated, i.e., that which exists in solution in the water, in combination with acids (carbonic, nitric, etc.), or in some other easily decomposable form.*

1. 500 c.c. (*i.e.*, half a litre) of the water are measured out, and placed within the boiling-flask.

2. If the water is acid, or even neutral, a little pure anhydrous sodium carbonate should be added so as to ensure alkalinity. The motive for this is primarily to enable the free ammonia to come away readily, since any acidity would exert a fixing influence upon it.

3. The boiling flask is then tightly connected by the cork with the condenser—*tightly* in order that uncondensed vapour shall not escape at this point. The Bunsen burner is next lighted, the flame applied to the flask, and *rapid* boiling is encouraged, care being had that "spurting" does not take place.

4. The water tap is turned to such an extent that the water, after circulating in the outer tube, returns in a small stream to the waste-sink.

5. A Nessler glass is placed so as to catch the distillate, and when sufficient of this is collected so as to reach up to the level of the 50 c.c. mark, a second glass is substituted, and then a third.

6. When three Nessler glasses are thus filled up to their 50 c.c. marks, a fourth is placed to catch the distillate, while 2 c.c. of Nessler's reagent are added to each of the three glasses. If these glasses be disposed upon the white porcelain slab from left to right in the order in which they received the distillate, the yellow colour created in each of them by the reagent will show a decrease in amount from left to right, since the first 50 c.c. collected will contain most "free ammonia," and the third the least.

7. The gas may be turned out, and the distillation stopped, if there is no colour in the third Nessler glass, or if it be *extremely* faint—since all the "free and saline ammonia" will then have come over. If, however, the colour is at all distinct in the last Nessler glass, a fourth must be collected and tested with 2 c.c. of the reagent; and even a fifth may be occasionally necessary. It is absolutely imperative that *all* the "free and saline ammonia" in the original 500 c.c. of water shall be removed, and it is rare in a drinking water that the 150 c.c. of distillate does not contain the whole of this.

8. The colour in the glasses is caused by the presence of ammonia, the amount of which must be determined by matching the colour in each glass by solutions containing a measured quantity of ammonia in the form of the ammonium chloride solution. Thus to match one of the glasses it is necessary to take a cylinder, and to deliver into it by a burette the amount of ammonium chloride standard solution (1 c.c. of which contains 0.01 milligramme of ammonia) that is

judged will be necessary to effect the match; the cylinder is then filled up to the mark with distilled ammonia-free water, and then 2 c.c. of Nessler's reagent are added. If the match is not correct, then a fresh comparison must be made with more or less of the standard solution, as the case may be. A very little experience will enable the operator to guess the amount of standard solution required to make the match, and to effect this with great rapidity.

It is pointed out by Wanklyn that it is not necessary to match each glass respectively, since three-quarters of the total amount of "free and saline ammonia" are contained in the first glass of distillate; that is to say, this ammonia comes off so remarkably readily and evenly, that on this account it is only necessary to compute the amount which the first Nessler glass contains, since this will constantly represent three-quarters of the total. Occasionally, however, there is a faint discrepancy between the amount thus calculated, and that obtained by matching each glass—this may be due to a difference in the degree of alkalinity of the water, and to the rate of boiling. On this account, and for the reason that the statement does not hold true with very foul waters, it is preferable in every case to estimate the amount of ammonia in each glass. The difference, however, is almost insignificant, so far as it can affect the opinion formed upon the sample.

In every case, when, after stirring with a clean glass rod, the colour is found to approach that in the distillate, wait about three minutes before deciding, since the colour deepens a little upon standing.

The presence and degree of coloration must always be judged by looking down through the depth of the water on to a white slab. Precaution must be taken that the bottoms of the glasses, and the upper surface of the slab also, are perfectly dry, as a thin layer of water intervening between

these diminishes materially the depth of colour, and thus leads to error in making a comparison.

9. Having thus effected a colour match by placing the two glasses side by side upon the white slab under exactly the same conditions of light access, the amount of ammonium chloride solution which has been used to effect this is noted, and the ammonia which this is equivalent to will be the amount of the "free and saline ammonia" in the glass of distillate.

*Example.*—150 c.c. of distillate were collected, the reagent added, and the last 50 c.c. distilled is found to contain no trace of ammonia. The whole of the "free and saline ammonia" in the 500 c.c. of water has therefore been collected.

It was necessary to add 3 c.c. of the standard solution of ammonium chloride to the comparison test-glass, in order to match the colour in the glass containing the first 50 c.c. of distillate, and 1 c.c. of the standard solution was required to match the colour in the second 50 c.c. of distillate.

The total amount then of "free and saline ammonia" in the 500 c.c. of water corresponds to the ammonia present in 4 c.c. of the standard solution.

But 1 c.c. of this standard solution contains 0.01 milligramme of ammonia.  $\therefore$  4 c.c. contains 0.04 milligramme of ammonia.

$\therefore$  there is 0.04 milligramme of "free and saline ammonia" in the 500 c.c. of water (or 500,000 milligrammes).

Wanklyn expresses the results of this process—dealing as we are with such small quantities—in terms of "parts per million," but for the sake of uniformity in the Report, the return of parts per 100,000 is preferred.

$\therefore$  there is 0.04 milligramme of "free and saline ammonia" in 500,000 milligrammes of water, or 0.008 parts per 100,000.

*The next step* in the process is to continue the distillation



more slowly after adding the recently boiled 100 c.c. of alkaline permanganate of potassium solution to the water left in the boiling flask ; to collect the distillate in three Nessler glasses, and to repeat the process of "Nesslerising" precisely as before. The ammonia estimated is here called "albuminoid ammonia," since it is derived from the breaking up of albuminoid and other nitrogenous organic matter by means of the alkaline permanganate at the temperature of boiling water. It is important to remember that in this case the ammonia comes over more slowly and much less evenly (the second Nessler glass sometimes containing as much as the first), so that grave errors (of under estimation) may be made by considering the first 50 c.c. of distillate to contain three-quarters of the total "*albuminoid ammonia*." Of course distillation must be continued, as in the former case, until *all* the "albuminoid ammonia" has been brought over.

*Example.*—It was necessary to distil over 200 c.c. in four Nessler glasses before all the ammonia had come over. The fourth glass of distillate had colour equal to that furnished by  $\frac{1}{2}$  c.c. of the standard solution ; the third to that created by 1 c.c. ; the second to 2 c.c., and the first to  $2\frac{1}{2}$  c.c.

$\therefore (\frac{1}{2} + 1 + 2 + 2\frac{1}{2}) = 6$  c.c. of the standard solution were required to match the colour furnished by the "albuminoid ammonia" in 500 c.c. of water.

But each c.c. of the standard solution = 0.01 milligramme of  $\text{NH}_3$ .  $\therefore 6$  c.c. = 0.06 milligramme of  $\text{NH}_3$ .

$\therefore$  there is 0.06 milligramme of  $\text{NH}_3$  ("albuminoid") in 500,000 milligrammes of water, or 0.012 milligramme in 100,000 of water.

*Conclusions to be drawn from the amounts estimated.*—Wanklyn's conclusions are generally accepted, viz., "when the albuminoid ammonia amounts to 0.05 part per million, then the proportion of free ammonia becomes an element in the calculation, but if 0.00, then it may be passed as organi-

cally pure, despite much free ammonia and chlorides. Free ammonia being very small, a water should not be condemned unless the albuminoid equals about 0.10," and, it may be added, if free ammonia is practically absent, the albuminoid may be allowed to considerably exceed 0.1—so as to meet the case of peaty waters.

A water is generally considered just within the border line of safety if the "free" and "albuminoid" ammonia are 0.005 and 0.008 parts per 100,000 respectively. Much "albuminoid" along with a small amount of "free" ammonia indicates vegetable contamination, and this indication gains further support if there is only a faint trace of chlorides and no excess of nitrates and nitrites. Much "free ammonia" and excess of chlorides, nitrates, and nitrites will denote animal pollution, though in those rare cases where a water is solely polluted by effluvia (arising from animal matter) there may be no excess of chlorine present.

Excess of "free" ammonia, unaccompanied by any corresponding excess of "albuminoid," may be due to the following circumstances:—

- (a) Some forms of organic pollution, as by urine.
- (b) The water has percolated a stratum containing a reducing agent (generally a reducing salt of iron) which has decomposed the nitrates and nitrites originally present in the water, as in the case of some deep well waters; or metal pipes, etc., with which the water comes in contact may effect this reduction.
- (c) The water has percolated strata in which some ammonia salt is present.
- (d) The sample is rain water, which sometimes contains ammonia in marked amounts when collected in towns.

The necessity of considering the amount of "free am-

monia," along with that of the "albuminoid," is best exemplified in the case of rain-water, in which "free ammonia" often exists in considerable quantities, and especially when collected in town districts, where it is washed out of the sooty atmosphere. In the rain collected in open country districts, faint traces of ammonia are always found, and some of it is combined with the nitric acid formed when electric discharges pervade the atmosphere.

The further steps of the analysis will always indicate the source of any excess of "free ammonia," and where it is derived from organic pollution the "albuminoid ammonia" will be also in excessive quantities.

The Nessler reagent will create the *faintest possible* evidence of a yellow colour in 50 c.c. of water not containing a suspicious amount of "free ammonia," when this is examined in a Nessler glass; if, however, this colour is distinctly apparent, the water is a very suspicious one. This forms a rough and reliable test of the freedom of a water from an excessive amount of "free ammonia," and to a great extent of its purity, but it must be borne in mind that such excess does not necessarily imply animal pollution, and a water may have recently acquired such pollution, and yet show but little "free ammonia." When, however, a marked amber tint appears when the Nessler reagent is added to the original water, such water is undoubtedly foul, unless it comes from a very deep well in the chalk and underlying greensand—in which case Wanklyn's process will detect practically no albuminoid ammonia.

As Professor Mallet points out, "the gradual evolution of albuminoid ammonia indicates the presence of organic matter in a fresh or comparatively fresh condition, whilst rapid evolution indicates that the organic matter is in a putrescent or decomposing state."

## NOTES UPON THE PROCESS.

Where the process is applied to extremely foul waters, or to sewage effluents, the degree of colour due to the ammonia in the first 50 c.c. will be so intense, that it will be impossible to match it with the standard solution, and in many cases a copious precipitate appears, and prevents comparison. In these cases, the distillate should be diluted with an equal bulk, and sometimes even with 5 or 10 times its amount, of distilled ammonia-free water; and when the amount of ammonia is estimated, allowance must be made for this dilution. It is well, therefore, if, when the Nessler reagent is added, a distinct amber tint appears, to consider the distillate too rich in ammonia to estimate without dilution, and thus save time and trouble.

All the materials used must be scrupulously clean, and if the condenser has not been used quite recently for the same process, or for distilling ammonia-free water, it is advisable to distil a little such water through, to ensure the absence of ammonia in the apparatus; care must also be had that no ammonia fumes are escaping—or have recently done so—into the atmosphere of the room.

The distilled ammonia-free water should always be redistilled quite recently, as, even when comparatively fresh, it is often found to contain minute traces of ammonia.

The boiling flask, if showing any fur from a previous analysis, should be cleansed with a little hydrochloric acid, and then washed with pure water until the washings are no longer acid.

The distillation adopted in the process adds materially to the delicacy of the operation of estimating the amount of the ammonia, for, *inter alia*, it separates the salts which the water contains, thus preventing the turbidity which generally arises when Nessler's reagent is added to undistilled water.

Sometimes while extracting the "albuminoid ammonia" the contents of the boiling-flask boil too violently, and "bumping" ensues; to obviate this a gentle shaking of the flask will often suffice, but in default a few fragments of freshly ignited tobacco pipe (as suggested by Mr. Duppa) afford an excellent remedy. The foulest waters, and those containing much saline matter, are most apt to bump, and it is highly important to prevent this, since uncondensed vapour thereby escapes at the distal end of the tube, and sometimes some of the water is shot over from the boiling-flask—both of which occurrences obviously vitiate the results; and when some of the water to which the alkaline permanganate has been added thus spurts over into the Nessler glass placed to collect the distillate it is of course impossible to "Nesslerise," since the distillate has a pink colour. There is no alternative then but to commence the process all over again with a fresh quantity of the sample.

When the "albuminoid ammonia" comes over so slowly (as in peaty waters) that almost all the water in the retort threatens to be used up, and in those rare cases where "the free ammonia" seems to hang about in small quantities, it is a good plan to adopt the measure (Rich) of repeating the process and "Nesslerising" the first 50 c.c., and then returning the rest of the distillate to the flask, and redistil it before "Nesslerising."

Strange to say, though the urea is decomposed by the boiling with the alkaline permanganate, its decomposition does not yield any ammonia, and this at first sight would seem a grave defect in the process. When, however, it is considered that this is, so far as has yet been shown, the only nitrogenous organic contamination to which a water is liable which does not, under the circumstances, yield ammonia, and that the urea in the urine becomes under ordinary conditions changed into ammonium carbonate almost as soon as it enters the



water, and as such is detected in the saline ammonia—the matter is not one of great moment. Moreover, it is not necessary that in the process the *total* nitrogen contained in organic matter should be evolved as ammonia, so long as that which *is* evolved gives an index which bears a pretty fixed and constant ratio to the total amount; so that from this index an empirical standard of purity can be formed. The process efficiently meets this requirement.

## CHAPTER XII.

## THE OXIDISABLE ORGANIC MATTER.

A CERTAIN quantity of any organic matter which may be present in water is always oxidisable, but unfortunately this amount varies with the nature of the organic pollution, and since it bears no constant ratio, its estimation furnishes no reliable index to the total quantity of such pollution present.

In the various attempts to estimate the oxidisable organic matter in water, advantage is taken of the well known chemical fact that in the presence of organic matter the permanganate of potassium, under favourable conditions, will part freely with its oxygen, until all the permanganate has become reduced to hydrated manganese dioxide, and that such change is denoted by the original pink colour which this salt gives to the water being replaced by one of a brownish hue.

Unfortunately this reaction of the permanganate ("Condy's fluid") has become only partially understood by the laity, among whom there is a wide-spread impression that any water which has been rendered a distinct pink by this salt, and allowed to stand for about a quarter of an hour, and which does not at the end of that time show any change of colour, is, as regards organic pollution, above suspicion. No better instance of the truth of the old adage "a little knowledge is a dangerous thing," could well be adduced, for such a test when the water is alkaline—as it often will be—will not detect such quantities of animal pollution as would place the water well within the category of a dangerously polluted one—and this for the reason that two essential conditions of the test are

ignored, viz., that the water should be rendered acid (to induce the permanganate to part with its oxygen), and also heated to a certain temperature (to encourage the same result).

The permanganate of potassium is in some respects an unsatisfactory salt to work with ; it does not, for instance, oxidise albuminous matters, nor does it affect creatin, sugar, gelatine, urea or fatty matters ; the standard solution of the salt which has to be employed is very unstable, and in consequence unreliable unless frequently renewed—on this account small amounts only must be made at a time.

The estimation of *oxidisable* organic matter is apt to furnish misleading results, for some forms reduce less permanganate than others, though they may be equally significant of danger, and comparatively harmless peaty waters absorb much more oxygen than waters dangerously polluted with animal matter.

In the face of these drawbacks, and possessing as we do in Wanklyn's process the means of making a far closer and more reliable estimation of organic matter, what is the value of the test? It is useful as a means of instituting comparisons between the purity of different waters, or of the same water at various times, and it frequently furnishes corroborative evidence of such value as to warrant its general performance.

It is very essential to appreciate, and always to bear in mind, the important fact that there are other substances which water is liable to contain which will reduce the permanganate, besides organic matter :—*i.e.*, nitrites, ferrous salts and sulphur compounds other than sulphates ; so that it is necessary to dispose of or account for these before attributing the reduction in the permanganate solely to oxidisable organic pollution. Since  $\frac{1}{4}$  grain to the gallon of iron can be detected by the chalybeate taste which it imparts to the water, in the absence of any such taste the presence of iron may be disregarded (more especially as existing in the form of a *ferrous*

salt. To get rid of the nitrous acid and sulphur compounds other than sulphates, it is necessary to boil the water after acidulation with sulphuric acid, for about twenty minutes. If iron is markedly present as a ferrous salt one may deduct from the total oxygen consumed the amount necessary to convert the iron into the ferric condition (112 parts of iron in a ferrous form will require to this end 16 parts of oxygen).

In the absence when tested for of any of these other reducing agents, the process can, of course, be at once commenced without any preliminary treatment. The late Dr. Tidy made some valuable improvements in the working details of the original processes; but those drawbacks inseparable from the use of the permanganate, etc., and which have been pointed out, of course remain. In his method a certain temperature is insisted upon, as well as the employment of definite amounts of certain reagents; but it remains for analysts to agree upon the terms in which the results shall be invariably expressed, and the period of time for which the test shall be applied, in order to avoid the confusion arising from such various returns as "oxygen absorbed in 15 minutes," "in 30 minutes," "one hour," "2 hours," and "4 hours."

The precise temperature employed during the process has been proved by experiment to be a more important factor than would be imagined; the amount of oxygen abstracted from the permanganate varies considerably at different temperatures.

If the water is kept too long, the quantity of oxygen absorbed frequently increases; this in most cases results from a reduction of nitrates by organic matter (bacteria, etc.).

#### TIDY'S MODIFICATION OF THE FORCHAMMER PROCESS.

##### **Reagents required:—**

1. A standard solution of the permanganate of potassium, 10 c.c. of which contain 1 milligramme of available oxygen,

made by dissolving 0.395 gramme of the pure salt in a litre of distilled water.

2. A solution of potassium iodide, made by dissolving one part of the pure salt in ten of distilled water.

3. Dilute sulphuric acid (1 in 3); a solution of the permanganate of potassium is dropped in until a faint pink tint remains after four hours at a temperature of 80 deg. F.

4. A solution of sodium hyposulphite, made by dissolving 1 gramme of the crystallised salt in a litre of distilled water.

5. A freshly prepared solution of starch, made by adding 1 gramme to 500 c.c. of cold distilled water and briskly boiling for five minutes, then let settle, and decant the almost clear supernatant liquid.

**Apparatus required:—**

1. Two thin glass stoppered bottles of about 400 c.c. capacity each.
2. Two large glass beakers.
3. Two graduated burettes.

**THE PROCESS.**

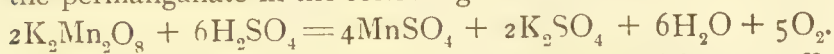
1. Pour 250 c.c. of the water into a thin glass bottle or flask closed at the top by a glass marble or stopper, and then place this inside a large stout beaker containing so much water that it rises up the sides of the bottle to well above the level of the contents. A water-bath is thus improvised.

2. Add 10 c.c. of the dilute acid, then apply heat until the 250 c.c. of the water in the bottle have reached a temperature of 80 deg. F., regulate the calibre of the flame, and remove it away from the centre of the under surface of the beaker until the heat applied is just sufficient to retain the water under examination constantly at about 80 deg. F.—this being a temperature which facilitates the parting of the oxygen from the permanganate of potassium, and its absorption by the organic matter present. As a very small flame is all that is required



to keep the water at the necessary temperature, it will be found convenient to use Fletcher's burner rather than the ordinary type of Bunsen.

3. Add to the 250 c.c. of water 10 c.c. of the standard solution of permanganate. Sulphuric acid liberates oxygen from the permanganate in the following manner:—



But here the amount of sulphuric acid added is not sufficient to make the permanganate part with its oxygen, but merely to assist it in doing so in the presence of the extra stimulus of oxidisable organic matter.

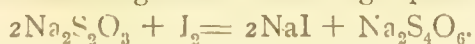
4. After two hours (or after four hours), take the bottle from the bath and proceed to estimate the amount of *undecomposed* permanganate it contains in the following manner:—

5. Add a drop or two of the solution of the iodide of potassium, stirring well with a clean glass rod, until the pink colour is entirely removed and a yellow one (due to free iodine) replaces it; *i.e.*, the undecomposed permanganate immediately reacts upon the iodide with the result that an amount of free iodine is liberated proportionate to the amount of undecomposed permanganate:—



The next step is to estimate the amount of this free iodine, and it is done in the following manner:—

6. Add by a graduated burette the standard solution of sodium hyposulphite until the yellow colour has nearly disappeared, *i.e.*, little free iodine remains; and so as to estimate the remainder with great precision, create the blue colour of the iodide of starch by adding a drop or two of the starch solution; then resume the addition of the standard solution of sodium hyposulphite until this blue colour has just disappeared. The reaction of the hyposulphite solution upon the free iodine is according to the following equation:—



7. If this process of discoloration has been properly performed, and if the necessary amount of hyposulphite solution has not been exceeded, a drop of the permanganate solution will suffice to restore the blue colour to the water.

8. The solution of hyposulphite is extremely liable to change, and it is therefore advisable to standardise it upon each occasion of trial by a control test as follows:—

250 c.c. of doubly distilled water are treated in precisely the same manner, and for the same time, as the water under examination, and the 10 c.c. of standard solution of permanganate should not, of course, be in any way affected in this case; so that when we come to estimate the free iodine in terms of the hyposulphite solution, the quantity of this necessary for the titration will be the amount which is equivalent to 10 c.c. of the standard solution of permanganate (containing 1 milligramme of available oxygen). The difference, therefore, between the amount of hyposulphite solution here required and that required to titrate the amount of free iodine liberated by the permanganate in the sample of water will indirectly represent the amount of permanganate decomposed.

*Example.*—The *distilled* water + 10 c.c. of permanganate used up 26.5 c.c. of the hyposulphite solution.

∴ 26.5 c.c. of the hyposulphite solution may be considered as equivalent to 10 c.c. of permanganate, or the 1 milligramme of oxygen which this will part with.

The *sample* water + 10 c.c. of permanganate used up only 16.5 c.c. of the hyposulphite solution, and therefore an amount of oxygen equivalent to  $26.5 - 16.5 = 10$  c.c. of hyposulphite solution has been taken up by the organic matter. But if 26.5 c.c. of hyposulphite solution is equivalent to 1 milligramme of  $O_2$ , then  $10 \text{ c.c.} = 0.377 \text{ milligramme of } O_2$ .

∴ 0.377 milligramme of  $O_2$  is taken up by 250 c.c. of water (250,000 milligrammes) or the organic matter in a hundred thousand parts of water required 0.15 parts of oxygen to oxidise it.

## NOTES UPON THE PROCESS.

The amount of permanganate added during the heating must in every case be sufficient to create a pink which remains distinctly permanent at the end of the heating. In some cases, therefore, of foul waters, it is necessary to make further additions of the permanganate solution, when careful note must of course be made of the *total* amount which has been employed.

At the end of the process, *i.e.*, after titration, the blue colour returns when the fluid has been exposed a few minutes to the air.

Two hours is quite short enough for the test to be of much value—since it is highly probable that it is mainly the *putrescent* organic matter which is *chiefly* oxidised in the first hour.

*Conclusions to be drawn from the amount estimated.*—In a *very pure* water the oxygen thus absorbed does not exceed about 0.05 parts per 100,000 after two hours; but a water cannot be classed as *suspicious* (by this test) unless it absorbs over 0.1 in two hours, and even in these cases no definite conclusion can be come to unless the nature of the organic pollution is roughly known, since a peaty water could not be judged as harmful which required twice this amount of oxygen to oxidise its organic matter; this is due to the fact that the marked amount of vegetable matter absorbs a great deal of oxygen from the permanganate—more so than an equivalent weight of most forms of animal matter; and waters may contain a considerable quantity of the former (as peat) and not be generally productive of ill-health.

## CHAPTER XIII.

## DR. E. FRANKLAND'S PROCESS.

A SHORT abstract only of this truly ingenious process is here appended, since it is too difficult and complex for any but trained chemists to perform, and it is generally found that Wanklyn's method attains to as true an estimate of the organic matter at the cost of far less trouble. A correct appreciation of the process is necessary, however, for the Public Health student, since the chemists of the London Water Companies and the Official London Water Analyst on behalf of the Local Government Board adopt the process, and send in their returns accordingly; moreover, the process was used in the important analyses performed for the Rivers Pollution Commissioners. The chief reason, therefore, for introducing it here, and offering a short abstract of the clever and elaborate process, is to enable the reader of such reports to understand the significance of the terms employed to express results.

The process is a favourite neither among health officers nor with the generality of analytical chemists, and it may be interesting and instructive to review the reasons of its unpopularity.

The reader will remember that it was pointed out on a previous page that the rationale of the method is as follows. When water is evaporated to dryness and the residue is burnt with the oxide of copper, the nitrogen and the carbon which result from the combustion of the organic matter can be collected and estimated as "organic nitrogen" and

"organic carbon." It is evident, however, that in every case it is necessary to consider and dispose of the nitrogen and carbon which may be originally present in the water, the former as ammonia and oxidised nitrogen, and the latter as carbonates and free carbonic acid.

The chief objections raised against the process are:—

- (a) Its cost. The expense of the mercury and apparatus is great (a description of the latter would entail many pages of print).
- (b) Its difficulty of performance except in the hands of trained and skilful chemists.
- (c) The fact that a large bulk of water has to be evaporated to dryness must ensure *some* amount of breaking up and dissipation of the less stable organic matter. In some cases it has been found that losses consequent upon this prolonged evaporation may approximate in their proportion to the total amount of organic matter which is estimated by the process.
- (d) The process is tedious and requires at least two days for completion.
- (e) Inferences are drawn from the ratio which "organic nitrogen" bears to "organic carbon," and the amount of "organic nitrogen" is less reliably estimated than that of the latter. Dr. Dupré points out that sea-water shows a ratio between the two worse even than is found in pure sewage.
- (f) The dangers of errors in working, and delays sometimes occasioned by the fracture of combustion tubes, are greater than that in any process employed in a water analysis.

In the face of these obvious drawbacks it is maintained—and justly so—that results are certainly no more precise and trustworthy than those obtained by Wanklyn's method; and as regards the opinion which these results enable one to form



upon the water, it *closely* coincides with that formed when the same water is analysed by Wanklyn's method.

#### THE PROCESS.

1. To a litre of water add 15—20 c.c. of a saturated solution of sulphurous acid, and boil briskly for a few seconds. This serves to decompose the carbonates present and expel carbonic acid, and to eliminate as nitric oxide the nitrogen in nitrates and nitrites; but if there is a large quantity of nitrates present 2 or 3 drops of acid solution of ferrous chloride should be added to facilitate their destruction. The water is thus freed of the nitrogen and carbon which is not present in the form of organic matter.

2. Evaporate the water to dryness in a dish protected from the access of dust and atmospheric ammonia. If the water originally contained no carbonates, 1 or 2 c.c. of a solution of sodium bisulphite must be added, in order to combine with the sulphuric acid, which if free would decompose the organic matter on concentration.

3. The residue is next intimately mixed, by means of a spatula, with pure oxide of copper in a finely powdered state; this is then suitably packed in a combustion tube with copper oxide and metallic copper, and placed in a combustion furnace for about an hour; then by means of a Sprengel pump the resulting gases are collected over mercury. These gases are carbonic acid, nitric oxide, nitrogen, and extremely rarely carbonic oxide. Occasionally, if the operation has been conducted too rapidly, sulphurous acid may also be present.

4. The mixture of gases is first freed of any sulphurous acid present by means of a concentrated solution of potassium bichromate; the absence of sulphurous acid being assured, the gases are measured volumetrically; the carbonic acid is absorbed by a strong solution of caustic potash, and the residual gases (consisting of nitrogen and a small quantity of

nitric oxide) are again measured. The gas is then exposed to a few drops of a saturated solution of pyrogallie acid to see if oxygen is present (which rarely happens from traces of oxygen given off by the cupric oxide, which pass so rapidly over the metallic copper as to escape absorption). If oxygen is absent a little is introduced to oxidise the nitric oxide to pernitric oxide, which is absorbed by the solution of caustic potash, and the excess of oxygen is absorbed by pyrogallie acid solution. The remaining gas is then measured, and consists of nitrogen.

The trace of carbonic oxide above referred to, especially liable to be present when the amount of carbon is large, and which rarely escapes complete oxidation by the copper oxide, may be removed by cuprous chloride.

Thus three volumetric estimations are made, *i.e.*, (A) of the total volume of carbonic acid, nitrogen, and nitric oxide; (B) of the volume of nitrogen and nitric oxide; and (C) of the volume of nitrogen. Therefore,  $A - B =$  the volume of carbonic acid and  $\frac{B - C}{2} + C =$  the volume of nitrogen.

By this process the purity of water is judged from a consideration of the *actual amounts* of organic carbon and organic nitrogen present, and their *relative proportions* to each other; and both a low quantity of each and a small relative amount of organic nitrogen is favourable to the water. Much carbon and little nitrogen is indicative of vegetable pollution, whereas, on the other hand, if the relative proportion of N and C is high the inference is that the pollution is largely of animal origin.

The Rivers Pollution Commissioners held that "a good drinking water should not yield more than 0.2 part of organic carbon, or 0.02 of organic nitrogen in 100,000 parts."

## CHAPTER XIV.

## OXIDISED NITROGEN (NITRATES AND NITRITES).

NITRATES and nitrites in a water represent, almost entirely, the oxidised nitrogen derived from nitrogenous organic matter (chiefly animal). Organic matter, by its putrefaction and decomposition, becomes ultimately reduced to its absolute elements, of which nitrogen is one, and this nitrogen combining with hydrogen first forms ammonia; hence when "free or saline ammonia" is found in large quantities in a water it generally affords evidence of the actual presence of a polluting organic source, such as raw sewage. As the water continues on its course and percolates soil and porous strata, the N acquires oxygen from the water, the air, and to a far greater extent, by the action of so-called "nitrifying organisms" in the soil, and thus first becomes partially oxidised to nitrous acid ( $\text{HNO}_2$ ), which combining with bases (commonly of calcium, and less often of sodium and potassium) forms *nitrites*; the presence of these, therefore, generally indicates organic pollution, which, if not actually present, must have been very recently so. The same causes continuing to act, the nitrous acid combines with more oxygen, and becomes nitric acid ( $\text{HNO}_3$ ), and *nitrates* of these same bases are formed until ultimately none of the original N may have escaped this further oxidation. The oxidising process cannot proceed beyond the formation of nitrates, and their presence thus indicates that owing to the favourable conditions met with, some, it may be *all*, of the organic matter has been thoroughly oxi-

dised into innocuous nitrates by the time the water had reached the point where it was collected.

If "the two ammonias" are very small in amount then practically the whole of the organic matter may be considered as thus purified; when this is not the case, however, purification has only been partially effected. When, as in some rare cases, the water in its subsequent flow meets with reducing agents in the strata (either inorganic or organic), the nitrates which have been built up may become gradually deoxidised, and by successive retrograde steps become reduced to the original ammonia again; but in these cases there is practically no albuminoid ammonia, oxidisable organic matter, etc., so that the large amount of free ammonia will not be taken as due to animal pollution.

"Their determination (nitrates and nitrites) is therefore a point of the greatest importance, for they indicate either a pollution of the water at some remote period with possibly dangerous ingredients, or the contamination of the water at the present time with partially or completely purified sewage. At any time, however, the purifying power of the filtering earth may be exceeded or overcome, and then the liquid filth may pass into the well with its dangerous ingredients unchanged and unpurified." (Louis C. Parkes, "Hygiene and Public Health," Fourth Edition).

It is necessary, however, in all cases where nitrates exist, before ascribing their origin to organic pollution encountered by the water, to preclude the possibility of their access from soluble nitrates in the strata permeated, since waters of great organic purity from the chalk, the oolite, the red sandstone and the Lias may contain marked traces.

When then the traces present have not been derived from such pollution (which will be animal for the most part, and if vegetable will be mainly derived from the dangerous fungi), the water will have permeated the chalk and red sand-

stone formations, etc.; there will therefore be considerable hardness and mineral residue, and the direct evidence of organic matter will also be of a negative character—such as little saline and organic ammonia, oxidisable organic matter, etc. A knowledge of these facts will at once enable a true estimate of the importance of the presence of nitrates to be made.

Next as to the inference to be drawn from the presence of nitrites; they either indicate the incomplete nitrification of ammonia, or the reduction of nitrates by mineral reducing agents or microbes; thus when they occur in shallow wells or rivers their presence should suffice to condemn the water for drinking purposes, but when they occur in deep well or spring water they *may* not denote present danger; for in the latter case they may result from the reduction of nitrates. Nitrites have of course a tendency to rapidly become nitrates in water, so that whereas a water may contain the latter without any evidence of the former, nitrates will always be found accompanying nitrites.

Nitrates and nitrites exist in very small quantities in waters vitiated by vegetable matter alone, and chiefly because vegetable decomposition yields comparatively little nitrogen and plant life removes nitrates and nitrites from a water; thus a polluted water subsequently exposed to plant life may, after a time, furnish in its oxidised nitrogen but very faint evidence of its previous pollution.

Dr. Frankland has classified all the inorganic nitrogen present in a water, *i.e.*, that contained in nitrates, nitrites, and free or saline ammonia—after deducting “the average amount of nitrogen present in rain water”—as evidence of “previous sewage contamination”; but it will be seen, from what has already been said, that a water from certain strata may take up soluble nitrates, which could scarcely be taken as evidence of previous sewage pollution; the rainfall, moreover, over differ-



ent districts often shows such varying amounts of ammonia, that "the average amount of nitrogen in rain water" is a very unsatisfactory factor to deal with.

#### QUALITATIVE TESTS FOR NITRATES.

The old brucine test in careful hands will detect extremely faint traces.

A few drops of a saturated solution of brucine are well mixed up with half a test-tubeful of the suspected water; then with the test-tube held well on the slant against a white background pure sulphuric acid is poured gently down its sides until it forms a distinct layer at the bottom of the test-tube. When the test-tube is brought to the vertical a pink zone is seen to occupy the line of junction between the mixture of brucine and water and the sulphuric acid; the pink is very transitory, however, and soon changes to a brownish-yellow, hence the necessity of having previously provided a white background in order that the colour shall not be missed.

If no coloured zone appears the test-tube should be gently shaken, not so as to mix its contents, but so as to bring more of the water and brucine in contact with the sulphuric acid; if the results are still negative nothing but very insignificant traces of nitrates can be present.

A more delicate mode still of applying the same test is to place 2 c.c. of the water in a perfectly clean platinum dish and evaporate to dryness. Then a drop of pure sulphuric acid is allowed to fall into the dish, and a minute crystal of brucine is added. A pink colour will appear with extremely faint traces (*i.e.*, 0.01 parts per 100,000).

The ordinary tests for nitrates are responded to equally by nitrites. To detect nitrates in the presence of nitrites Piccini's test is valuable:—

Some pure urea is added to the water, and the mixture is slowly emptied into another solution of urea in dilute sul-

phuric acid. The effect of this is that all the nitrous acid is decomposed, and after the reaction has had time to complete itself, the presence of nitric acid may be tested for. After applying the starch test, and noting by the absence of any immediate blue coloration the complete removal of nitrous acid, a little finely divided zinc is added, and heat applied, when any nitric acid present is reduced to nitrous acid, and a blue colour results.

#### QUALITATIVE TESTS FOR NITRITES.

The old starch test for nitrites is sufficiently reliable and delicate, when carefully performed, for most purposes; but there must be no sulphuretted hydrogen in the water. It consists in the addition of a little clear starch solution and a drop of a solution of potassium iodide to some of the water in a test-tube. Dilute sulphuric acid is then added, and in the presence of nitrites a dark blue tint appears *immediately*, *i.e.*, nitrous acid is liberated by the sulphuric acid, it then reduces the potassium iodide, leaving the iodine free to combine with the starch and to form the *blue* iodide. The test should be performed at the lowest possible temperature, and an instant reaction must take place, for nitrates give similar results after standing awhile.

A more reliable and delicate test, however, is that of Griess:—5 grammes of meta-phenylene-diamine are dissolved in 100 c.c. of water (the solution when not perfectly colourless must be decolorised, *i.e.*, by animal charcoal, and it is a good plan to keep some of the charcoal in the bottle); the solution is afterwards slightly acidified with dilute sulphuric acid. If 1 c.c. of this be added to 100 c.c. of the suspected water in a Nessler glass, and this is gently warmed and placed upon a white porcelain slab, a pale yellow to an orange tint appears, according as the nitrites are present in smaller or larger amounts.

The solution of meta-phenylene-diamine must not be acidified too strongly, and in every case after applying the test the glass should be covered up, and half an hour should be allowed for the reaction to take place. The colour does not fully develop in less than half an hour, and this fact must be allowed for in the process of titration (*vide* Quantitative Estimation).

The most delicate appreciation of nitrites is made by acidifying a large bulk of the water with acetic acid, and then testing a little of the distillate that first comes over; but if the water contains sulphuretted hydrogen this must first be separated by means of a little well-washed carbonate of lead, and then filtering.

#### THE QUANTITATIVE ESTIMATION OF NITRITES.

The estimation is commonly based upon Griess' test, and the degree of colour thus created is matched by adding a standard solution of potassium nitrite (1 c.c. = 0.01 milligramme of  $N_2O_3$ ) to a similar quantity of distilled water which has been otherwise treated in like manner to the suspected water, in another Nessler glass.

The standard solution of potassium nitrite is made of the required strength by dissolving 0.406 gramme of pure silver nitrite in hot distilled water, and decomposing it with a slight excess of potassium chloride. This is allowed to cool, and the solution is then made up to one litre; allow the chloride of silver to settle, and dilute each 100 c.c. of the clear supernatant liquid again to one litre; one c.c. of this liquid contains 0.01 milligramme of  $N_2O_3$ , and this  $\times 0.37 = N$ . The solution should be kept in the dark.

The quantity of  $N_2O_3$  present is estimated by taking several cylinders containing amounts of standard nitrite solution varying say from 0.02 to 0.2 milligramme of  $N_2O_3$  in 100 c.c. of distilled water, and the reagent must be added to

the water and comparison cylinders at the same time, since the colour deepens upon standing.

*Example.*—It took, say, 8 c.c. of the standard nitrite solution to create in the comparison Nessler glass the same tint of colour which the nitrous acid present in the water sample furnishes, and therefore the amount of  $\text{N}_2\text{O}_3$  in the sample of water is equivalent to that contained in 8 c.c. of the standard solution.

But 1 c.c. of this = 0.01 milligramme of  $\text{N}_2\text{O}_3$ .

Therefore 8 c.c. = 0.08 milligramme of  $\text{N}_2\text{O}_3$ .

Therefore there are 0.08 milligramme of  $\text{N}_2\text{O}_3$  in 100 c.c. (or 100,000 milligrammes) of water, or 0.08 parts of  $\text{N}_2\text{O}_3$  in 100,000 parts of water.

The amount of  $\text{N}_2\text{O}_3 \times 0.37$  will represent "*the nitrogen in nitrites*" = 0.029 parts per 100,000.

The starch, iodide and zinc reaction may also be taken advantage of as a means of making a quantitative estimation.

#### THE QUANTITATIVE ESTIMATION OF NITRATES AND NITRITES.

The most convenient method—while at the same time as reliable as any of the many other processes to the same end—is that known as the *copper-zinc couple process*, by which all the oxidised nitrogen in nitrates and nitrites is reduced to ammonia by a wet copper-zinc couple. The amount of the ammonia thus obtained can be readily estimated as in Wanklyn's method, and taken as an index of the nitric and nitrous acid from which it was originally derived.

##### **Apparatus required:—**

1. A small wide-mouthed glass stoppered bottle.
2. A boiling-flask.
3. A condensing apparatus.
4. Nessler glasses.

**Reagents required :—**

1. Thin zinc foil—clean and bright.
2. A solution of cupric sulphate, about 2 per cent.
3. Occasionally some pure sodium chloride and oxalic acid.
4. The reagents for Griess' test for nitrites.
5. The Nessler reagent, and standard chloride of ammonium solution.

**THE PROCESS.**

1. A wet copper-zinc couple is prepared by taking a piece of thin well-crumpled zinc foil—clean and bright—measuring about three inches by three (of about three grammes weight), and covering this with a concentrated solution of copper sulphate.

Very quickly the bright surface of the zinc loses its metallic appearance and becomes tarnished with a black adherent coating of metallic copper which envelops the foil. As soon as this has thoroughly formed, and generally from 3 to 5 minutes will suffice, the zinc with its copper coat is removed—or the coating becomes pulverulent and falls away. It is then well washed with distilled ammonia-free water. Finally it is placed in a thoroughly clean 8-ounce glass stoppered bottle, with a wide mouth in order that it may take the metal.

2. 100 c.c. (or 250 c.c.) of the water under analysis are poured in so as to cover the "couple," and the bottle is tightly stoppered and left all night in a warm place (65 to 85 deg. F.).

With very soft water a trace of sodium chloride should be added (about 0.1 gramme), and with very hard ones a small quantity of pure oxalic acid to precipitate the lime.

3. On the following morning a small measured quantity of the water should be removed and tested for nitrous acid by Griess' test; the absence of this acid proves the completion



of the process, and its presence demands that the reaction should be given more time to complete itself in.

In the absence of nitrites the remainder of the water is decanted into a 500 c.c. measuring flask, the bottle is well washed out with some of the ammonia-free distilled water, the washings are also added to the 500 c.c. flask, and then this is filled up to about the mark with more ammonia-free water.

4. The water is then distilled until all the ammonia present has come over. This is then Nesslerised as in Wanklyn's method, and the *nitrogen* present is calculated from the ammonia formed thus:—

The molecular weight of ammonia =  $N(14)H_3(3) = 17$ , therefore  $N = \frac{14}{17}$  of the ammonia estimated.

Of course the amount of free ammonia originally present in the water, and which has already been estimated by Wanklyn's method, must be deducted from the total ammonia estimated by this process.

*Example.*—The 500 c.c. furnish 0.25 milligramme of ammonia, all of which must have been yielded by the 100 c.c. of sample less the small measured quantity removed for the purpose of testing for nitrites; this amounted to 25 c.c.

There is therefore 0.25 milligramme of ammonia in 75 c.c. of sample water, or 0.33 parts per 100,000.

But by Wanklyn's method the water showed 0.008 parts per 100,000 of free ammonia.

Deducting this amount therefore ( $0.33 - 0.008 = 0.322$ ) there are 0.322 parts of ammonia due to nitrates and nitrites in 100,000 parts of the water sample.

The results are expressed in terms of "*nitrogen as nitrates*," or as "*nitrogen as nitrates and nitrites*" in those cases where nitrites are also present. Nitrogen has been seen to form  $\frac{14}{17}$  of ammonia, therefore there are  $\frac{14}{17}$  of  $0.322 = 0.265$  of "*nitrogen as nitrates*," or of "*nitrogen as*

*nitrates and nitrites*," as the case may be, in 100,000 parts of water.

If in those cases where nitrates coexist with nitrites it should be desired to estimate the nitrogen of the *nitrates* alone, the nitrogen yielded by *nitrites* must be deducted. This is rarely, if ever, done now for any practical purpose; but the matter is a simple one. The water has been found by Griess' method to contain nitrogen as nitrous acid to the extent of 0.029 parts per 100,000.

The amount of nitrogen, therefore, which must be deducted on account of that furnished by nitrites = 0.029, and  $0.265 - 0.029 = 0.236$ , *i.e.*, the amount furnished by nitrates alone in 100,000 parts.

#### NOTES UPON THE PROCESS.

The ammonia thus furnished is generally in considerable quantities, and its colour cannot on this account be directly matched by the chloride of ammonium solution; it can best be estimated by dilution of the distillate with distilled ammonia-free water, as previously recommended (*vide* "Wanklyn's Method").

*Conclusions to be drawn from the amount estimated.*—The interpretation to be placed upon the presence of nitrites has already been sufficiently discussed. A large amount of nitrates will indicate previous pollution, either distant and remote or near and recent. When the "nitrogen in nitrates" exceeds 0.2 part per 100,000, suspicion is justified in those cases where the strata may be excluded as the source from which the water may have derived such nitrogen, but where such a source cannot be excluded an amount exceeding 0.5 would be regarded as suspicious, for it is rare that more than 0.3 part per 100,000 is derived from sources other than animal pollution. No mere limit, however, can be accepted

for all waters; the amount of oxidised nitrogen must be considered in conjunction with the results of the other processes that furnish evidence of contamination.

#### QUICK METHODS OF ESTIMATING THE "OXIDISED NITROGEN."

**The phenol-sulphuric acid method** of estimating nitrates is not quite as delicate as the copper zinc couple process, but the results can be very much more rapidly arrived at. The lesser delicacy of the process results in a very slight error of under-estimation.

The **reagents required** are:—

1. Phenol-sulphuric acid, made by mixing 8 vols. and 1 vol. respectively, of pure sulphuric acid and pure phenol, heating for several hours at 212 deg. F., and then diluting to three times the original bulk with distilled water. Preserve in a tightly stoppered bottle.
2. A standard solution of potassium nitrate (0.7214 gramme to the litre), each c.c. of which contains 0.1 milligramme of nitrogen.
3. Ammonia solution.

#### THE PROCESS.

20 c.c. of the water sample and 10 c.c. of the standard nitrate solution are each placed in a clean platinum dish, and evaporated just to dryness. 3 c.c. of the phenol-sulphuric acid, followed by a couple of drops of pure sulphuric acid, are then run into each of the dishes, which are subsequently allowed to remain on the water-bath for about three minutes.

The contents of the two dishes are separately placed into two Nessler glasses, and the dishes are washed out with ammonia solution—cautiously, to avoid loss from the spurting that takes place. The washings of each dish are placed in the same Nessler glasses which originally received their re-

spective contents, and then ammonia solution is added to each glass until effervescence ceases. The contents of the glasses are then filtered if necessary, and made up to 50 c.c. with distilled water. The Nessler glass containing the nitrate of the standard solution assumes a distinct yellow colour (due to the formation of ammonium picrate), and the contents of the other Nessler glass are coloured, more or less, according to the amount of nitrates in the 20 c.c. of water sample.

By transferring measured quantities from the deeper coloured liquid (which will of course be that containing the potassium nitrate standard solution) into other Nessler glasses, which are again filled up with distilled water to their marks, a match is effected; that is to say, it is learnt how much of the deeper coloured liquid is required, when diluted to the 50 c.c. mark with distilled water, to match the tint created by those nitrates which are furnished by the water sample in the other Nessler glass; or the darker solution may be poured into a measuring glass and successive additions of water made until 50 c.c. poured into a Nessler glass is found to be of the same tint as the 50 c.c. with which it is compared.

Suppose that as much as 5 c.c. are required of the 50 c.c. of the darker coloured liquid (containing the 10 c.c. of standard nitrate solution). Then of course ( $\frac{5}{50}$  or  $\frac{1}{10}$  of the 10 c.c. =) 1 c.c. of the standard solution are required, to match the colour created by nitrates, in the 20 c.c. of water sample.

But 1 c.c. of the standard solution contains 0.1 milligramme of nitrogen; therefore there is 0.1 milligramme of "nitrogen in nitrates" in 20 c.c. (20,000 milligrammes) of the water, or there is 0.5 milligramme of "nitrogen in nitrates" in 100,000 milligrammes of water, or 0.5 part per 100,000.

### The Indigo method.

This method proceeds upon the following principles:—Free nitric and nitrous acids are liberated from their combinations by a large quantity of concentrated pure sulphuric acid; these liberated acids are then measured by means of a weak solution of indigo in water containing 4 per cent. by volume of sulphuric acid—the exact strength of which as compared to a weak standard solution of potassium nitrate is accurately determined. From the amount of indigo solution it is necessary to add to a measured quantity of water before the blue colour of the indigo is quite discharged, the equivalent amount of potassium nitrate is estimated, and this  $\times 0.14$  represents the amount of “nitrogen in nitrates and nitrites” originally present in the measured bulk of water.

1 c.c. of the indigo solution is added to 20 c.c. of water, and a volume of strong sulphuric acid equal to that of the water and indigo solution (*i.e.*, 21 c.c.) is gently poured into the water. When the blue colour of the indigo commences to fade, stir, and run in more indigo solution till the blue colour remains permanent. Supposing 4 c.c. of the indigo solution are required to this end, then another 20 c.c. of the water are taken, 4 c.c. of the indigo solution are added, and 24 c.c. of acid, and the mixture again stirred, when decoloration commences; and thus the test is renewed until the precise quantity of indigo solution required by 20 c.c. of water to furnish a faint blue tint after a volume of acid corresponding to the indigo solution and water has been added is ascertained; the amount of nitrate solution which this is equivalent to is calculated, and this  $\times 0.14 = N$ .

If 20 c.c. of water require more than about 10 c.c. of indigo solution, it should be diluted.

A strong solution of indigo is made by digesting 2 grammes of pure sublimed indigotin in about 10 grammes of fuming



sulphuric acid for several hours, and then diluting to a litre with distilled water and filtering.

In standardising an indigo solution 10 c.c. of the standard nitrate solution should be taken, and it is ascertained by experiment how much of the indigo is required to effect a permanent faint blue tint, when an amount of sulphuric acid, equal to that of the nitrate and indigo solutions combined, is rapidly added and mixed, the flask being transferred to a calcium chloride bath at 28.4 deg. F. The estimated excess of indigo solution required to furnish the faint permanent tint must of course be deducted.

The process gives good results, and it is rapidly accomplished, but it cannot be relied upon when waters are distinctly contaminated with certain kinds of organic matter; the standardising of the indigo solution is, moreover, very troublesome. The best directions for performing the process with the minimum of error of experiment will be found in papers by Warrington in "The Chemical News," xxxv., 45, and "The Journal of the Chemical Society," 1879, 578.

## CHAPTER XV.

## EXAMINATION OF THE SEDIMENT.

**Chemical.**—Since the estimation and examination of the total solids have only included those solids which are in solution, it will be desirable in some instances to make a chemical examination also of the sediment. In these cases it is necessary to collect a fair quantity of the deposit, and the most simple method by which this can be effected is the following : After well shaking remove a litre of the sample, place it in a large conical flask, cover up and set aside for several hours ; then decant off as much of the supernatant water as it is safe to do without running any risk of disturbing the deposited matter ; that remaining behind in the flask should then be passed through a filter.

The sediment thus collected should be well-washed with pure water, dried, and the whole carefully incinerated at as low a temperature as possible. The mineral residue is then analysed.

Wynter Blyth's tube is a convenient instrument for collecting water sediments ; as seen in fig 29, it is similar in appearance to a huge pipette which is capable of holding about a litre of water. A small glass cell fits over the small lower extremity of the tube, into which the deposited matter collects after a time. By the insertion of the long rod-shaped stopper, the cell and the sediment within it can easily be removed.

If the suspended matter is so light that it will not settle, drops of the water must be examined under the microscope,

and if it is inorganic, the tests denoted on pages 127-128 should be applied under the cover glass. But it must be extremely rare that any visible suspended matter does not deposit in great measure after the lapse of several hours.

Where, however, a sediment forms from the sample of water, any analysis which fails to take advantage of the important positive evidence which a **microscopic examination** affords, will miss a most important step—since this alone in some cases may suffice to condemn a water, by detecting elements which denote danger. It is mainly, after all, the *nature* of the organic matter which denotes present danger, and by a microscopical examination of the water, direct evidence of this can in many cases be acquired. Some of the sediment, then, should be collected from another glass by means of a small pipette, in the manner adopted with urine sediments. It is then transferred to several glass slides, cover-

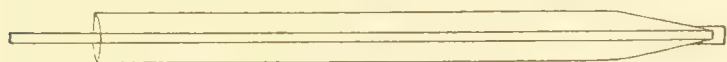


FIG. 29.—Wynter Blyth's tube for collecting sediments.

glasses are applied, and the examination by the microscope commenced, any excess of water upon the slide being removed by clean blotting-paper.

The various forms of animal and vegetable life, and of inanimate organic and inorganic material, are best sought after by commencing with the 1 inch power, next passing on to the  $\frac{1}{4}$  inch power, and then prosecuting the examination further by means of the  $\frac{1}{12}$  inch oil immersion lens and an Abbé's condenser. Further steps are necessary in the examination of bacteria, for which the reader is referred to the last chapter in the book.

Before giving a list of animate organisms which the naked

eye or microscope may disclose, it must be pointed out that those detected by the  $\frac{1}{4}$  inch power do not (with few exceptions) convey danger in themselves, but that it is rather their presence which implies such danger—denoting as it does that some pabulum, and possibly

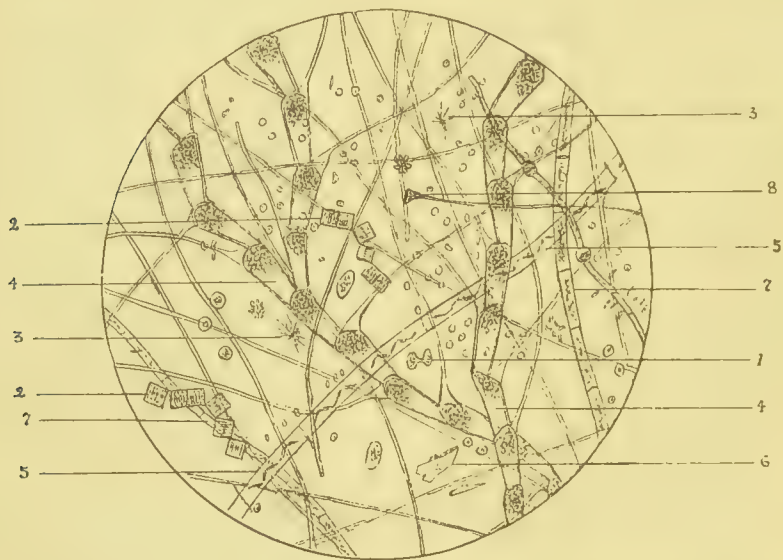


FIG. 30.—Showing the sediment of a pond-water, a sample of which was collected in the early Spring ( $\times 250$ ). Drawn by A. E. Evans, M.B.

1. A desmid; 2. *Tabellaria floccosa* (Diatomaceæ); 3. *Actinophrys*; 4. A confervoid growth; 5. A vegetable fibre showing spiral cells; 6. Silicious particles; 7. *Conferva*; 8. *Gomphonema*.

Various forms of minute unicellular plants are seen scattered about the "field."

a dangerous one, accompanies them, upon which they are capable of feeding and existing. Though this pabulum may be vegetable organic material or its products—as it frequently is, since they mostly exist in stagnant pond water in greatest numbers—the presence of these organisms in abundance, bearing as they generally do a ratio to their food supply, will be often sufficient in itself to condemn the water as

containing at least an injurious amount of vegetable pollution. The fact that animal and vegetable life have powers of purifying the water is beside the question of a sanitary analysis, their very presence denotes impurity, and with the attainment of purity they mostly disappear.

The higher and macroscopical types of animal life, such as water-fleas, water bears, amphipoda and isopoda, etc., broadly speaking denote less danger than the lower and more minute forms (bacteria, amœbæ, infusoria). The former are generally associated with suspended matter in waters that are not likely to be used for drinking purposes on account of their contamination being obvious to the senses; while the latter are more generally associated with dissolved organic matter in waters possessing excellent physical characters.

The most suspicious elements are those which point directly to sewage contamination, and next to these, those which point indirectly to such contamination. The latter will be found in hair, wool, cotton and linen fibres, reddish-brown globular masses, etc., the former in those objects enumerated on pages 131-132.

The greatest evidence of danger is afforded by the discovery of large numbers of bacteria and fungi, anguillulæ, and infusorians.

#### MATTER WHICH MAY BE FOUND BY A MICROSCOPICAL EXAMINATION OF WATER.

##### I. INANIMATE.

(a) *Mineral*.—Sand and flint particles generally have a sharp and angular outline, though often somewhat rounded from rolling and attrition. A drop of hydrochloric acid let to run under the cover-glass has no effect upon them.

Clay and marl (alumina-silicate) particles are amorphous, and very minute, and are unaffected by hydrochloric acid.



Chalk particles are likewise amorphous, mostly somewhat larger than those of clay and marl, and generally rounded in outline. A drop of hydrochloric acid dissolves them, *i.e.*, causes them to disappear with effervescence.

Iron peroxide forms a reddish-brown amorphous debris, soluble, like the chalk, in hydrochloric acid, and blued when a drop of the solution of ferrocyanide of potassium is allowed to run under the cover-glass.

Mica forms thin fine films of very irregular outline.

(b) *Vegetable*.—Parenchymatous cells; the dotted ducts of the vessels; spiral vessels, or spiral fibres; pieces of the cuticle with the vegetable “hairs” still adhering; pollen.

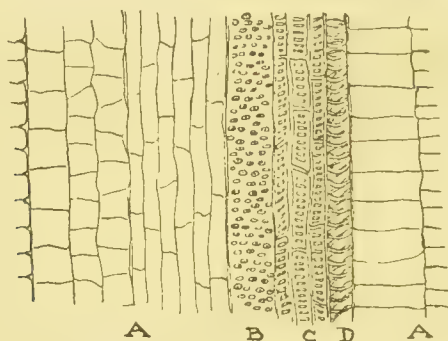


FIG. 31.—Vegetable tissue showing : A. Vegetable parenchyma; B. A pitted vessel; C. A scaliform vessel; D. A spiral vessel.

Woody fibres; fragments of leaves, etc.

Starch cells, of wheat, potato, etc.

Macerated paper. Linen and cotton fibres.

Vegetable matter mostly appears as dark flattened structureless masses (opaque), or frequently only as debris—when the diagnosis is attended with great difficulty; if, however, any spiral vessels, or fibres, or dotted ducts can be distinguished, these will always point to the nature of any obscure debris, etc., with which they are associated.

Dark masses from soot may be present in the water.

(c) *Animal*.—Hairs; feathers; down.

Wool or silk fibres.

Striped muscular fibres; fat cells and crystals; connective tissue; epithelial scales; shreds of mucous membrane.

Scales from the Lepidopterous insects (moths, etc.), and wings, legs, etc., of other insects.

Reddish-brown globular masses are sometimes justly attributed to sewage pollution.

## II. ANIMATE.

(a) *Vegetable*.—Minute forms of vegetable life mostly belong to the class of cryptogamous (*i.e.*, non-flowering) plants, and contain chlorophyll, and are in consequence green. They may be divided into :—

1. *Small and microscopic fungi*, which, with moulds, represent the lowest forms of vegetable parasites. They may be present as spores, sporangia, or as mycelia.

Both bacterium termo and sarcina ventriculi present familiar instances of these forms.

Beggiatoa alba has been badly named "the sewage fungus," but any water containing sulphates is capable of supporting the fungus, quite independently of the source from which such sulphates are derived.

2. *Numerous forms of algæ*, ranging in size from those only visible by high microscopic powers, to those visible with the naked eye. Of these there are many families :—The volvocineæ, of which volvox is the type, comprise the lowest vegetable forms of minute unicellular organisms; the oscillatoria (*vide* plate iii.) exhibit a pendulum-like motion; the confervaceæ (*vide* plate iii.), etc.

Protococcus pluviialis is an interesting instance of an algoid plant which can live in the atmosphere, and which is often found in pure rain-water.

Desmids and diatoms would all be included in this class. The algæ, when in considerable quantities, furnish a dark green repulsive appearance to the water, and may give rise to diarrhœa, and when they die and decay the water acquires an offensive taste and smell; otherwise they are not of much hygienic importance.

(b) *Animal*:—1. *Protozoa*. (a) Rhizopoda. Amœba will be recognised by its characteristic amœboid movement. Actinophrys is another common and familiar form; and polyps shows a very low type of structure. In spongilla fluviatilis (the fresh water sponge) the animal substance is spread over a network of spicules.

(b) Infusoria. Paramœcium, vorticella, coleps, and euglena viridis, are all common types.

Stentor is among the largest of this class, and is so named from the trumpet-like shape of the body.

2. *Cœlenterata*.—Hydra is a common type of this sub-kingdom.

3. *Annulosa*.—This sub-kingdom embraces:—

(a) Crustacea including the Amphipoda,\* Isopoda† and Branchiopoda‡.

Both cyclops quadricornis, gammarus pulex and daphnia pulex are familiar types of this class; in the latter the antennæ act as oars and propel the little animal through the water by a series of short springs or jerks; they assume a red

\* The Amphipoda are sessile-eyed malacostracans. Their bodies are compressed laterally, the eyes are immobile, and their feet are directed partly forwards and partly backwards.

† The Isopoda possess sessile eyes and a depressed body, and the feet are of equal size and move in the same direction.

‡ The Branchiopoda are so-called because their branchiæ or gills are situated on the feet. The head is not distinct from the thorax, which is much reduced in size.

colour in summer, and when in swarms they give a bloody tinge to the water.

(b) Arachnida, including water bears (the tardigrada), etc.

(c) Insecta. Either in the larval, pupal, or adult forms.

4. *Annuloida*.—This sub-kingdom embraces the scolecida, and includes turbellaria, rotifera (or wheel-animalcules), tæniada, næmatoidea, anguillulæ (water-worms).

5. *Mollusca*.—Including polyzoa, siphonida, etc.

The various *parasites* which may be conveyed through the medium of drinking water are:—

The segments and eggs of tape-worms (*Tænia Solium*, *T. Mediocanellata*, *T. Echinococcus*, and *Bothriocephalus Latus*); the Guinea-worm (*Dracunculus Medinensis*); small leeches (*Hirudinidæ*); the Round-worm (*Ascaris Lumbricoides*); the Thread-worm (*Oxyuris Vermicularis*); *Bilharzia Hæmatobia*; *Dochmius Duodenalis*; *Tricocephalus Dispar* (the Whip-worm); *Filaria Sanguinis Hominis*; the filarial stage of *Distomum Hepaticum* (the Liver-fluke of sheep).

These latter may be found in either embryonic or adult stages of development.

From out of this confusing mass of objects what material may be selected as affording direct evidence of human sewage contamination? *i.e.*, which are those objects which can rarely, except along with sewage, gain access to the water? They are either (1) substances which from their indigestibility commonly leave the body in the fæces; (2) substances which may do so when digestion is interfered with; or (3) the various animal parasites which infest the human gastro-intestinal tract.

Under the first heading would be embraced such substances as:—Various connective tissue elements, fat cells and crystals, muscle fibres, some vegetable cells and starch grains, etc. Under the second heading shreds of mucous

membrane, mucus, epithelial cells, gall stones, quantities of all kinds of food in a semi-digested state, etc. The third heading includes *T. Solium*, *T. Mediocanellata*, *Bothrioccephalus Latus* (either as eggs or segments); *Ascaris Lumbricoides*, *Oxyuris Vermicularis*, *Tricocephalus Dispar* (ova or mature forms).



## CHAPTER XVI.

## GASES IN WATER. THE OPINION UPON A WATER ANALYSIS.

It is generally known that water can absorb almost all gases or vapours coming in contact with it, and this with great readiness—for it is the aeration of water which gives to it the pleasant taste and sparkling appearance which is so general. The degree of aeration—as has been already pointed out—affords no evidence of the water's purity or impurity, since the foul water of a shallow polluted well is frequently markedly aerated, whereas the pure water collected from great depths in the chalk (notably in the case of many artesian wells) is sometimes poorly so.

Rain water when thoroughly aerated contains about 20.73 c.c. of gases per litre, *i.e.*, nitrogen 13.08, oxygen 6.37, and carbonic acid 1.28 c.c.

A water naturally highly charged with carbonic acid gas is the well known Appollinaris water.

To ascertain if free **carbonic acid** exists in the presence of bicarbonates, a solution of 1 part rosolic acid in 500 parts of 80 per cent. alcohol (and to which baryta water has been added until it begins to acquire a red tint) may be used;  $\frac{1}{2}$  c.c. of this, added to 50 c.c. of water, causes distinct reddening in the absence of free  $\text{CO}_2$ .

For estimating the amount of carbonic acid, which is free and as bicarbonate, the following method (after Pettenkofer) will be found very convenient and exact.

Place 200 c.c. of the water into a perfectly dry flask, add 10 c.c. of a neutral and nearly saturated solution of calcium chloride, 5 c.c. of a saturated solution of ammonium chloride,

and then 35 c.c. of baryta water—standardised immediately before testing with oxalic acid. The chloride of calcium will decompose any alkaline carbonate or any other alkaline salt whose acid would be precipitated by lime or baryta; the ammonium chloride will avoid the precipitation of any magnesia present.

The flask is then tightly closed with an india-rubber stopper, well shaken, and allowed to stand for 12 hours—and preferably in hot water for the first hour or so.

The flask contains altogether 250 c.c. of fluid; 100 c.c. of this amount are carefully decanted so as not to disturb any of the precipitate, and then divided into two portions of 50 c.c. each. The free uncombined baryta is then estimated in both portions—for control purposes—by means of a standard solution of weak oxalic acid (2.8636 grammes pure crystallised acid to the litre; 1 c.c. = 1 milligramme  $\text{CO}_2$ ), using phenolphthalein as indicator.

The exact number of c.c. of oxalic acid solution employed in the 50 c.c. of fluid is then multiplied by 5—since there were 250 c.c. of fluid altogether in the flask—and this number is deducted from the number of c.c. of standard acid which is required to exactly neutralise 35 c.c. of the original baryta water. The difference shows the baryta precipitated by  $\text{CO}_2$ , free and as bicarbonate, in the water.

*Example.*—50 c.c. of the fluid from the flask required 2.5 c.c. of oxalic acid solution to exactly neutralise it.

$\therefore 2.5 \text{ c.c.} \times 5 = 12.5 \text{ c.c.}$  are required for the whole 250 c.c. of fluid; 30 c.c. of the standard acid are required to neutralise the 35 c.c. of the original baryta water.  $30 \text{ c.c.} - 12.5 \text{ c.c.} = 17.5 \text{ c.c.}$ , which represents the baryta precipitated by  $\text{CO}_2$ .

But 1 c.c. of the standard acid = 1 milligramme of  $\text{CO}_2$ .

$\therefore 17.5 \text{ c.c.} = 17.5 \text{ milligrammes of } \text{CO}_2$  which were present in 200 c.c. of water; or 87.5 milligrammes per litre.

**Sulphuretted hydrogen** most frequently gains access to water by the putrefaction of organic substances, or indirectly from industrial waste matters. It may be estimated while in the water in the following manner:—

Take a 500 c.c. flask and run in 10 c.c. of centi-normal iodine solution, and then run in a measured quantity of the water until the yellow colour of the free iodine disappears ( $I_2 + H_2S = 2HI + S$ ); 5 c.c. of starch solution are then added and the addition of the iodine solution cautiously continued until a blue colour just begins to show itself. Each c.c. of iodine solution used has decomposed 0.17 milligramme of  $H_2S$ . The slight excess of  $I_2$  required to produce the blue colour is trivial, but it may be estimated and deducted by titrating back with sodium hyposulphite.

In addition to the innocuous gases upon which the aeration of a pure water depends, *i.e.*, nitrogen, oxygen, and carbonic acid, it is obvious that water may at the same time take up noxious gases (such as the constituents of coal gas), or those which, though not noxious in themselves, yet—inasmuch as they are generally the products of organic putrefaction and decomposition—indicate the presence of danger, such as sulphuretted hydrogen, ammonia, marsh gas ( $CH_4$ ) and olefiant gas ( $C_2H$ ), etc.

Some waters issuing as springs in the vicinity of volcanoes are charged with sulphurous acid.

The estimation of the nitrogen and carbonic acid can serve no practical hygienic ends, as no conclusions of value can be drawn from the results, interesting as they are from a chemical point of view; but the estimation of oxygen is of some importance, for it gives a clue as to the power that the water possesses of destroying organic matter and some of the low forms of organic life, and it becomes rapidly diminished in oxydising the C, H and N of any putrescent organic matter present.

## THE ESTIMATION OF THE OXYGEN DISSOLVED IN WATER.

Dr. Thresh has devised a very satisfactory process for this estimation, one that is quickly performed and easy of execution. The following description is an abridgment of the author's paper as it appeared in the Journal of the Chemical Society for March, 1890.

When dilute sulphuric acid and the iodide of potassium are added to water containing a nitrite the amount of iodine liberated varies with the length of time during which the mixture is exposed to the air. If air be carefully excluded there is no increase in the amount of iodine set free after the first few minutes, and if the water be previously boiled and allowed to cool in an atmosphere of hydrogen or coal gas, still less iodine is liberated ( $2\text{HI} + 2\text{HNO}_2 = \text{I}_2 + 2\text{H}_2\text{O} + 2\text{NO}$ ).

When oxygen has access to the solution the nitric oxide acts as a carrier, and more hydrogen iodide is decomposed ( $2\text{HI} + \text{O} = \text{H}_2\text{O} + \text{I}_2$ ).

We have simply, therefore, to add to a known quantity of the water a definite amount of sodium nitrite, together with excess of potassium iodide and acid, avoiding access of air, and then to determine volumetrically the amount of iodide liberated. After deducting the proportion due to the nitrite used, the remainder represents the oxygen which was dissolved in the water and in the volumetric solution used.

The following are the reagents required:—

1. Solution of sodium nitrite and potassium iodide.

Sodium nitrite...     ...     0.5 gramme.

Potassium iodide     ...     20.0 grammes.

Distilled water     ...     100 c.c.

2. Dilute sulphuric acid. Pure sulphuric acid, 1 part;  
distilled water, 3 parts.

3. A clear or fresh solution of starch.

4. A volumetric solution of sodium thiosulphate. Pure crystals of thiosulphate, 7.75 grammes; distilled water to 1 litre. 1 c.c. corresponds to 0.25 milligramme of oxygen.

The apparatus is used in the following manner:—

The bottle A being cleaned and dry, the perforated bung is inserted, the burette charged, and the tube B fixed in its place. E is connected with the gas supply. The tube D

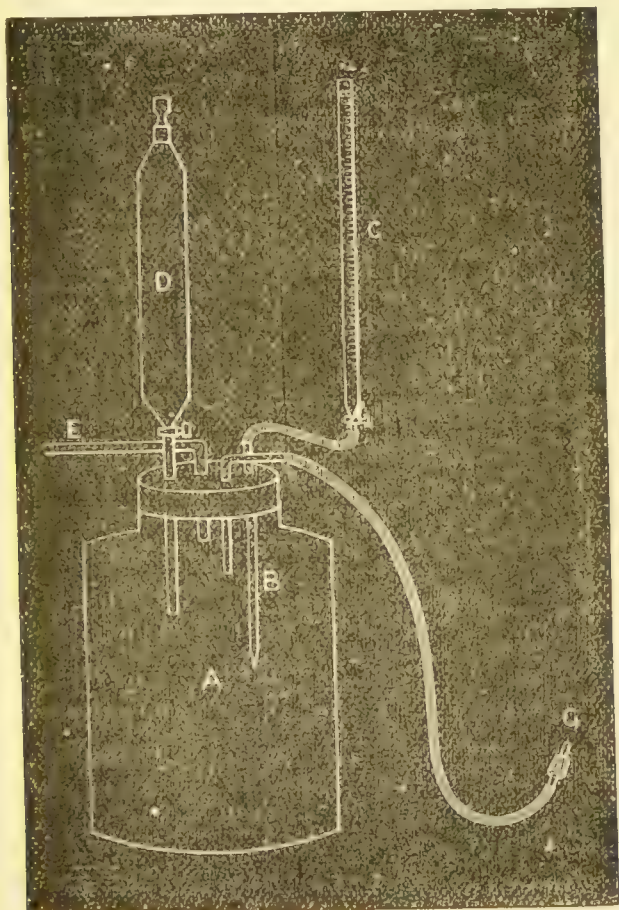


FIG. 32.—Apparatus for Thresh's process.

is filled to the level of the stopper with the water to be examined, 1 c.c. of the solution of sodium nitrite and potassium



iodide added from a 1 c.c. pipette, then 1 c.c. of the dilute acid, and the stopper instantly fixed in its place, displacing a little of the water, and including no air. If the pipette be held in a vertical position with its tip just under the surface of the water, both the saline solution and the acid, being much denser than the water, flow in a sharply defined column to the lower part of the tube, so that an infinitesimally small quantity (if any) is lost in the water which overflows when the stopper is inserted. The tube is next turned upside down for a few seconds for uniform admixture to take place, and then the nozzle is pushed through the bung of the bottle, and the whole allowed to remain at rest for 15 minutes, to enable the reaction to become complete. A rapid current of coal gas is now passed through the bottle A, until all the air is displaced, and the gas burns at G with a full luminous flame. The flame is now extinguished, the stopper of D removed, and the cork G rapidly inserted in its stead. On turning the stopcock, the water flows into the bottle A. The stopcock is turned off, the cork G removed, and the supply of gas regulated so that a small flame only is produced when this gas is ignited at G. Thiosulphate is now run in slowly from the burette C, until the colour of the iodine is nearly discharged. A little solution of starch is then poured into D, and about 1 c.c. allowed to flow into the bottle by turning the stopcock. The titration with thiosulphate is then completed. After the discharge of the blue colour, the latter returns faintly in the course of a few seconds, due to the oxygen dissolved in the volumetric solution; after standing about two minutes, from 0.05 to 0.1 c.c. of thiosulphate must be added to effect the final discharge. The amount of volumetric solution used must now be noted. This will represent  $a$ , the oxygen dissolved in the water examined, +  $b$ , the nitrite in the 1 c.c. of solution used, and the oxygen in the acid and starch solution +  $c$ , a portion of the dissolved

oxygen in the volumetric solution. To find the value of  $a$ , it is obvious that  $b$  and  $c$  must be ascertained. This can be effected in many ways, and once known does not require redetermination, unless the conditions are changed.

To find the value of  $b$  :—

Probably the best plan is to complete a determination as above described, and then, by means of the stoppered tube, introduce to the bottle in succession 5 c.c. of nitrite solution, dilute acid, and starch solution. After standing a few minutes, titrate. One fifth of the thiosulphate used will be the value required.

To find the value of  $c$  :—

This correction is a comparatively small one, and admits of determination with sufficient accuracy if we assume that the thiosulphate solution normally contains as much dissolved oxygen as distilled water, saturated at the same temperature. Complete a determination as above described, then remove the stoppered tube, and insert a tube similar to that attached to the burette, and drop in from it 10 or 20 c.c. of saturated distilled water, exactly as the thiosulphate is dropped in. Allow it to stand a few minutes and titrate. One-tenth or one-twentieth of the volumetric solution used, according to the number of c.c. of water added, will represent the correction for each c.c. of volumetric solution used. Call this value  $d$ . (It is taken as 0.31.)

Let  $e$  be the number of c.c. of thiosulphate used in an actual determination of the amount of oxygen in a sample of water.

$f$  = the capacity in c.c. of the tube employed—2 c.c., the volume of reagents added.

$g$  = the amount of oxygen in milligrammes dissolved in 1 litre of water ; then

$$g = \frac{1000}{4f} (e - b - ed.)$$

With a tube made to hold exactly 250 c.c., the most convenient quantity to use,  $\frac{1000}{4f}$  becomes unity, and

$$g = e - b - ed.$$

A few of the results got by Dr. Thresh are as follows :—

Source of Water.	f. Amount of water employed.	e. Thiosul- phate required.	e—b—ed.	g. Milli- grammes O per litre.
1. Spring water ...	322'0	12'35	9'87	7'66
2. Rain water... ...	322'0	13'05	10'55	8'19
3. Shallow well water	322'0	11'35	8'90	6'91
4. Rain water... ...	322'0	12'95	10'45	8'11
5. Distilled water } shaken with air }	322'0	16'00	13'40	10'40

A water containing nitrites will require the amount of the nitrous acid to be determined if the utmost accuracy is required (a water containing 1 part  $\text{HNO}_2$  in 1,000,000, will affect the results + 0.17 milligramme of oxygen per litre, 94 parts of the acid corresponding to 16 of oxygen). Where nitrites are present in sufficient quantity to interfere, the amount may be determined by any of the ordinary processes.

Evidence of some of the other gases may be obtained by heating the water to 110 deg. F., in the manner already described when treating of "smell"; or frequently sulphuretted hydrogen and ammonium sulphide may be discovered in very small quantities by the addition of a solution of lead acetate, when the water darkens. In those cases a solution of the nitro-prusside of sodium (a few drops) will distinguish between sulphuretted hydrogen and ammonium sulphide, for

it furnishes a violet purple colour with ammonium sulphide, but remains unchanged if sulphuretted hydrogen alone be present.

Should, however, a more careful and delicate investigation be desirable, the gases may be liberated and collected by

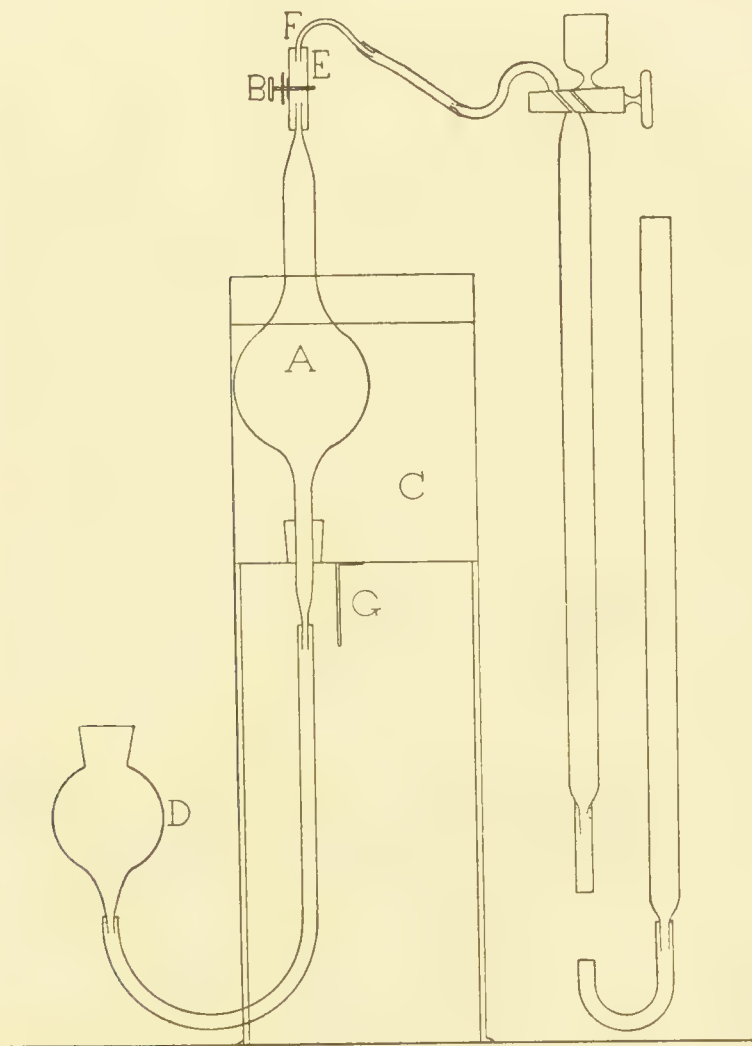


FIG. 33. — Harvey's apparatus for the extraction of gases dissolved in water.

Harvey's method, and estimated on principles set forth in "Air Analysis."

The apparatus for the extraction of gases dissolved in water, devised by Mr. Sidney Harvey, is simple, useful, and reliable; it requires little attention while working, there are no corks and but few connections, and the water and evolved gases do not leave the vessel until the end of the operation.

The nature of the apparatus is shown in fig. 33. The two tapering necks of the glass vessel A are from 13 to 14 inches long, the globular part being  $3\frac{1}{4}$  or  $3\frac{1}{2}$  inches in diameter. The upper neck has a short length of small bore pressure tubing, securely tied, and furnished with a screw clamp, B. The apparatus is used as follows:—The exact capacity of A from end to end is first ascertained; it is then filled with the water to be examined, the clamp B is closed, and the vessel carefully fixed upright in a water-bath standing on legs 12 inches high and having an opening in the bottom closed by a perforated cork. The clamp B is now opened, and about  $\frac{1}{3}$  of the water allowed to run into a measured vessel—the remainder being the amount experimented on. The lower end of the spindle has now one end of a two feet piece of small bore pressure tubing slipped over it and secured; and this tubing has a mercury reservoir, D, suitably supported, at its other end.

Mercury is now poured into the reservoir, the clamp B is again opened, and the air, together with any bubbles, driven out, the water being allowed to follow to the upper end of the rubber tube, E. After closing the clamp a nitrometer having a bent capillary tube, F, affixed to the beak, is filled with mercury, and this is forced to the end of the capillary tube, which is now thrust into the top of the rubber tubing and secured. The reservoir is lowered and the clamp cautiously opened in order to run a little mercury over sufficiently far to reach the lower end of the capillary tube; the clamp is



closed, the bath filled with cold water, and heat applied—G representing a metal screen to protect the glass from the flame.

Under the diminished pressure the water in the globe soon boils vigorously, and without bumping, and the expelled gases collect in the upper stem. After two hours the reservoir may be raised, the clamp B opened, and the gases passed into the nitrometer, taking care not to admit any water. The clamp is again closed, the reservoir lowered, and the operation continued to collect any further traces of gas. By raising the reservoir any residual gas may be driven completely into the nitrometer, where it can be measured, and its constituents separated by absorption.

A ball of caustic potash moistened with water is introduced into the graduated tube containing the gas, by means of a piece of platinum wire, on which it is cast. When there is no further diminution noted in the volume of the collected gases the bulb is replaced by another, and when absorption has ceased a dry bulb is inserted. This is removed after an hour, and the volume of the residual gas is noted. The loss in volume by this treatment represents carbonic acid and also, if present, sulphuretted hydrogen; the latter can be determined in the water in the manner already indicated and subtracted. The residual gases generally consist of oxygen and nitrogen. The oxygen is absorbed by introducing a ball of papier maché attached to the end of a platinum wire, and moistened with a concentrated alkaline solution of pyrogallate of potassium; the ball is replaced by another if necessary, and the gas is finally dried by a ball of caustic potash. The loss in volume sustained by the treatment with the alkaline pyrogallate represents the oxygen absorbed, and the residual nitrogen is then measured. All the gases must be reduced to the standard temperature and pressure.

Though ammonia will always exist in small quantities, when there is marked evidence of its presence, or when sul-

phuretted hydrogen (generally most evident in the summer months), ammonium sulphide, or the constituents of coal gas are present, then the water should be regarded with the greatest suspicion.

While sulphuretted hydrogen may, however, be derived from mineral sulphides, or from reduction of mineral sulphates (which reduction is frequently effected by organic matter and living organisms—such as *beggiatoa alba*), and the presence of ammonia may be due to reducing agents acting upon nitrates, yet these sources are very rare as compared with the danger yielding ones.

Good examples of waters charged with sulphuretted hydrogen from harmless sources are to be found at Harrogate and Aix-la-Chapelle notably. Some waters from the clay have a distinct odour of sulphuretted hydrogen, derived from the tiny particles of iron pyrites which enter into the composition of such clay.

#### ANALYSIS OF MINERAL WATERS.

The examination of mineral waters should be conducted on precisely the same lines as those of an ordinary water analysis—that is to say, an effort must be made to ascertain the freedom of the water from dangerous organic and metallic contamination.

Artificial “mineral waters” consist of water into which carbonic acid is forced under pressure:—Lithia water is, in addition, charged with lithia; potass water with potassium bicarbonate; soda water is commonly sold without the addition of any soda, but when such is added it is usually to the extent of 10 grains of bicarbonate to the pint.

Natural mineral waters are generally the purest. Those which are chalybeate mostly contain the iron in the form of ferrous carbonate, held in solution by excess of carbonic acid, such as those at Tunbridge, Spa, and Cheltenham. Instances

of alkaline waters naturally charged with carbonic acid, and containing sodium carbonate and bicarbonate are found at Carlsbad, Ems, Malvern, Nieder-Seltzers, and Vichy. At Harrogate and Aix-la-Chapelle waters are found naturally charged with sulphuretted hydrogen; those waters which possess a marked aperient action generally owe their properties to either magnesium sulphate, as at Epsom and Leamington, or to sodium sulphate, as at Cheltenham and Scarborough. In Central Wales there is a deep spring containing nine parts of barium chloride per 100,000.

All artificial mineral waters should be tested for lead, iron, copper, zinc, and arsenic; each of these metals has been found in samples of soda-water, etc., to which it has gained access either by the apparatus used, or by the improper washing of the carbonic acid. Sometimes very impure water is used; on this account it is desirable that efficient supervision should be exercised over their manufacture.

Lemonade and ginger-beer are two beverages especially liable to contain traces of lead, derived from the apparatus, or, in the case of the former, from the impure tartaric acid employed.

In natural mineral waters a careful analysis of the ash from a large bulk of water must be made, and the following acids and bases should be especially sought for:—

- (a) *Bases*.—Potassa, soda, lithia, baryta, strontia, magnesia, alumina, protoxide of iron, manganese, and the oxides of copper, zinc, lead, antimony, cobalt, and nickel.
- (b) *Acids, etc.*—Phosphoric, sulphuric, silicic, carbonic, boric, arsenic, arsenious; chlorine, bromine, iodine, fluorine, sulphuretted hydrogen.

Fresenius gives a good scheme for analysing the ash of a water residue.

The ash procured from a large bulk of water, at a dull red

heat, is first divided into three portions. 1 is examined for phosphoric acid, 2 for fluorine, and 3 is divided by repeated boilings with water and subsequent washings and filtration into (a) a residue, and (b) a solution.

(a) is tested for calcium carbonate, magnesium carbonate, silicic acid, hydrated ferric oxide, baryta, strontia, alumina, oxide of manganese, and titanitic acid. Some of the residue is further sub-divided by treatment with hydrochloric acid into (aa), that insoluble in hydrochloric acid, *i.e.*, silicic acid, sulphates of the alkaline earths, titanitic acid, and (bb), that soluble in hydrochloric acid, *i.e.*, alumina, manganese, iron, baryta, strontia.

(b). The alkaline solution contains the salts of the alkalies, and usually also magnesia and traces of lime. But some must be concentrated by evaporation, and also tested for boric acid, and the residue from a further portion divided into two parts, one to be tested for nitric acid, iodine, and bromine, the other and smaller part for lithia.

The sodium and potassium may be estimated in the following manner :—Take a litre of water and add to it a few drops of baric chloride solution in order to precipitate the sulphuric acid ; boil with pure milk of lime to precipitate the magnesia, iron, and phosphoric acid (if present) ; filter, concentrate the filtrate ; add ammonia, ammonium carbonate, and a few drops of ammonium oxalate, again filter, and evaporate the filtrate to dryness. Next ignite to expel ammoniacal salts, but do so with extreme care, to avoid volatilisation ; treat the residue with a little water, filter if necessary, acidify with hydrochloric acid, and evaporate to dryness in a weighed platinum dish. The alkalies may then be weighed as chlorides. The potash may be separated by a 10 per cent. solution of platinum chloride ; the precipitate is washed with alcohol, and collected on a tared filter, dried at 212 deg. F., and weighed, when the potash is obtained as  $\text{PtCl}_2\text{KCl}$ , and this  $\times 0.19308$

represents the weight of  $K_2O$ , and by 0.3056 the equivalent amount of  $KCl$ .

By deducting the weight of potassium chloride from the sum of the mixed chlorides, the remainder may be taken as sodium chloride which  $\times 0.531 = Na_2O$ .

Lithia may be estimated by adding to a litre of water, phosphate of soda and sufficient solution of soda to leave the reaction alkaline; evaporate to dryness, treat the residue with water, filter, add an equal volume of ammonia. Very minute quantities of lithia separate as tribasic phosphate. The precipitate is soluble in hydrochloric acid, and the solution remains clear when supersaturated with ammonia, but on boiling yields a heavy crystalline precipitate of phosphate.

In the case of wells, specimens of the water at various depths should be collected.

The whole of the dissolved gases must be collected and analysed.

The temperature of an immersed thermometer (at the time of collecting) should also be taken, and the specific gravity should be ascertained—compared with that of distilled water taken as 1000. If the sample is collected from a well the thermometer should be let down in a stout glass bottle; this will come up filled with the water, and a reading of the thermometer when surrounded by the water can be taken.

#### THE OPINION UPON A WATER ANALYSIS.

It will be seen from what has already been written that there are many considerations to be carefully weighed together before any definite opinion can be formed upon the presence or absence of organic pollution in any given water sample.

There is no necessity, however, in those cases where it is desired to form a *rapid conclusion* as to whether a water



may be safely drunk for the time being, to subject it to a complete and thorough analysis, since the quantitative estimation of the "two ammonias," the chlorine and the nitrates, together with a qualitative examination for nitrites and the poisonous metals, will here suffice ; but it is desirable that a water analysis should be as inclusive and complete in most cases as it is possible to make it.

In expressing an opinion upon the analysis, it is very desirable to adopt terms which, while leaving no doubt in the mind of anyone as to the opinion held upon the water, shall not be *too* dogmatic ; and thus it will always be better to say that "the water shows no evidence of harmful organic pollution," than to employ such a precise statement as "*there is* no organic pollution" ; and the results of a single examination in the case of well, river, and pond water, must not be considered sufficient evidence to enable one to pronounce aught but the fact that *the sample* examined is that of a good water. In order to commit oneself to a statement which would imply that the source from which the water is derived is a *constantly* pure one, it will be necessary to examine many samples at different seasons, and under different conditions of rainfall, drought, etc.—which furnish important factors in determining the different degrees and characters of the fouling which may obtain.

It would be of great assistance to some medical officers of health if they carefully constructed "water standards" for their districts. Such would be prepared by selecting the *purest* water in each locality, and would form a ready and reliable means of detecting the smallest amount of additional impurity which may have gained access to the supply of any particular household, the water of which it may be desirable to examine. For instance, a water sample may well contain 2.5 parts per 100,000 of chlorine without any suspicion being aroused ; but say that the average of the pure water of the

locality is 1.5 parts, then the excess becomes important evidence of "added chlorine," the presence of which would be most significant.

It will be appropriate here to recapitulate the amounts which when found in the most important stages of the analysis of water would generally arouse suspicion as to the safety of the water.

*"Free" ammonia and "albuminoid" ammonia.*—If the "albuminoid" ammonia exceeds 0.005 parts per 100,000 the "free" should not be above this amount, but if the former is considerably below this then much "free" ammonia may not signify danger; conversely the "albuminoid" ammonia may reach to 0.01 part per 100,000 if the "free" is much below 0.005, and if there is practically no free ammonia, *i.e.*, below 0.002, then the albuminoid ammonia may be allowed to considerably exceed 0.01.

*The oxidisable organic matter examined by Tidy's process at 80 deg. F.*—The oxygen absorbed must exceed 0.1 parts per 100,000 in two hours, or twice this amount in peaty waters, before its amount can be regarded as suspicious.

*Chlorine.*—More than 1 part per 100,000, if not accounted for by the strata permeated, would arouse distrust.

*The oxidised nitrogen.*—More than 0.2 parts per 100,000 of nitrogen in nitrates and nitrites excites suspicion. The faintest trace of nitrites will generally condemn waters collected from no great depth.

*The poisonous metals.*—More than  $\frac{1}{20}$  grain of lead and  $\frac{1}{10}$  grain of copper to the gallon should suffice to condemn. Zinc and iron should certainly not exceed  $\frac{1}{4}$  grain. The faintest trace of arsenic condemns.

*The total solids.*—These should not exceed 50 parts per 100,000, but of course much depends upon their constitution. In no case, except in peaty waters, should there be much

charring on ignition, nor should fumes or odour ever be appreciable during this process.

*The hardness* should not exceed 30 parts per 100,000, and the greater the proportion of "temporary" to "permanent hardness" the better. More than 10 parts of permanent hardness would be regarded with disfavour.

*Sulphates*.—Should not exceed 10 parts of  $\text{SO}_3$  per 100,000.

*Phosphates*.—More than 0.05 part  $\text{P}_2\text{O}_5$  per 100,000 arouses suspicion.

## CHAPTER XVII.

## WATER SAMPLES.—ANALYTICAL SCHEMES.—SEWAGE EFFLUENTS.—ALKALIMETRY AND ACIDIMETRY.

*Sample I.*

## A VERY PURE WATER.

(i.e., Showing amounts which, when not exceeded, denote great purity in all the steps of the analysis).

*Physical Characters.*—Pleasant taste. Well aerated. Clear and bright. No marked colour. No odour. No sediment.

Reaction . . . . .	Neutral.	
Saline Ammonia . . . . .	0.002	} parts per 100,000.
Organic Ammonia . . . . .	0.002	
O <sub>2</sub> absorbed from Permanganate . . . . . (in 2 hours at 80° F.)	0.04	
Total Solid Matters . . . . .	12	
(a) Volatile . . . . .	2	} No appreciable discoloration. No smell or fumes given off.
(b) Fixed . . . . .	10	
(c) Appearance on ignition . . . . .		
Total hardness . . . . .	10	} grains per gallon.
(a) Temporary . . . . .	7	
(b) Permanent . . . . .	3	
Chlorine . . . . .	1.5	
Equivalent to Common Salt . . . . .	2.5	
N as Nitrates and Nitrites . . . . .	0.01	
Poisonous Metals . . . . .	nil.	
Microscopical Examination of Sediment.	—	

*Note.*—As has been pointed out, if the oxidisable organic matter is of a vegetable nature, the 0.04 parts per 100,000 may be exceeded in even a very pure water.

### *Sample II.*

#### A FOUL AND DANGEROUS WATER.

*Physical Characters.*—Unpleasant taste and odour (on heating). A faint dirty (yellow or brown) colour. Turbid. Sediment. Poorly aerated.

Reaction . . . . .	Alkaline.	
Saline Ammonia . . . . .	0.01	} parts per 100,000.
Organic Ammonia . . . . .	0.015	
O <sub>2</sub> absorbed from Permanganate . . . . .	0.2	
(in 2 hours at 80° F.)		
Total Solid Matters . . . . .	60	
(a) Volatile . . . . .	25	
(b) Fixed . . . . .	35	
(c) Appearance on ignition . . . . .	{ Considerable char- ring, fumes and odour given off	
Total hardness . . . . .	30	
(a) Temporary . . . . .	15	
(b) Permanent . . . . .	15	
Chlorine . . . . .	8	} grains per gallon.
Equivalent to Common Salt . . . . .	13.2	
N as Nitrates and Nitrites . . . . .	0.8	
Poisonous Metals . . . . .	{ Dangerous amounts of Pb, Cu, or Zn.	

*Microscopical Examination of Sediment:*—Low forms of animal and vegetable life, such as bacteria and fungi; and organic debris or evidence of human excretal contamination in epithelium, cotton fibres, undigested particles, paper, etc.



*Notes.*—Water polluted by sewage *emanations* (gases and vapours) shows an absence of this excess of chlorine and nitrogen as nitrates and nitrites; but other evidences of organic pollution are marked, and especially the “free ammonia.” Microscopically, abundant evidence of bacteria is forthcoming.

0.2 parts of  $O_2$  absorbed would not necessarily indicate danger, if the organic pollution were vegetable.

### Sample III.

#### A SUSPICIOUS WATER.

*Physical Characters.*—Slightly coloured (shades of yellow or brown).

Not perfectly clear. No marked taste or odour. Slight sediment.

Poorly aerated.

Reaction . . . . .	Alkaline.	
Saline Ammonia . . . . .	0.006	} parts per 100,000.
Organic Ammonia . . . . .	0.012	
$O_2$ absorbed from Permanganate . . . . .	0.16	
(in 2 hours at 80° F.)		
Total Solid Matters . . . . .	40	
(a) Volatile . . . . .	15	
(b) Fixed . . . . .	25	
(c) Appearance on ignition . . . . .	{ some charring, no fumes or smell.	
Total hardness . . . . .	25	
(a) Temporary . . . . .	15	
(b) Permanent . . . . .	10	
Chlorine . . . . .	4	} grains per gallon.
Equivalent to Common Salt . . . . .	6.6	
N as Nitrates and Nitrites . . . . .	0.3	
Poisonous Metals . . . . .	{ nil (except faint traces of iron).	

Microscopical Examination of Sediment:—A few low forms of animal and vegetable life, of questionable origin and significance.

*Sample IV.*

## RAIN-WATER.

*(As collected generally in country districts).*

*Physical Characters.*—No colour apart from that of pure water. Quite clear. Pleasant taste. No odour. Well aerated. No sediment.

Reaction . . . . .	Slightly alkaline.	
Saline Ammonia . . . . .	0'0025	} parts per 100,000.
Organic Ammonia . . . . .	0'0005	
O <sub>2</sub> absorbed from Permanganate . . . . .	0'025	
(in two hours at 80° F.)		
Total Solid Matters . . . . .	3'0	
(a) Volatile . . . . .	1'5	
(b) Fixed . . . . .	1'5	
(c) Appearance on ignition . . . . .	no charring, etc.	
Total Hardness . . . . .	0'75	
(a) Temporary . . . . .	0'25	
(b) Permanent . . . . .	0'5	
Chlorine . . . . .	0'25	} grains per gallon.
Equivalent to Common Salt . . . . .	0'41	
N as Nitrates and Nitrites . . . . .	0'015	
Poisonous Metals . . . . .	nil	
Microscopical Examination of Sediment.		—

*Notes.*—In towns the reaction is slightly acid, from the sulphurous acid in the atmosphere; and the water is a little different in other respects owing to further impurities taken up, such as soot, sulphur compounds, and ammonia.

That rain-water which is collected in country districts after long periods of continuous rainfall affords the purest possible *natural* water.

*Sample V.*

## A TYPICAL PEATY "UPLAND SURFACE" WATER.

*Physical Characters.*—The colour is generally yellowish-brown, but peat may furnish various shades of green, yellow, and brown,—or there may even be no colour at all. Not quite clear (from fine suspended matter). No marked taste or odour. Aeration slight.

Reaction . . . . .	Distinctly acid.	
Saline Ammonia . . . . .	0'0015	} parts per 100,000.
Organic Ammonia . . . . .	0'015	
O <sub>2</sub> absorbed from Permanganate . . . . .	0'220	
(in two hours at 80° F.)		
Total Solid Matters . . . . .	10	
(a) Volatile . . . . .	7	
(b) Fixed . . . . .	3	
(c) Appearance on ignition . . . . .	{ Some darkening, faint sweet odour	
Total Hardness . . . . .	5	
(a) Temporary . . . . .	1	
(b) Permanent . . . . .	4	
Chlorine . . . . .	1	} grains per gallon.
Equivalent to Common Salt . . . . .	1'6	
N as Nitrates and Nitrites . . . . .	0'009	
Poisonous Metals . . . . .	nil	

*Microscopical Examination of Sediment.*—Shows vegetable debris, fibres, cells and parenchyma, and frequently minute forms of vegetable life (*vide* Chapter on Water Sediments).

*Note.*—In many "peaty waters" the "organic ammonia" and the "oxygen absorbed" will be found to much exceed the amounts given above, which constitute the mean of many such waters analysed.

*Sample VI.*

## A SPRING WATER FROM THE CHALK.

*Physical Characters.*—No marked colour. No odour. Very palatable.

Well aerated. Quite clear. No sediment.

Reaction . . . . .	Alkaline.	
Saline Ammonia . . . . .	0'001	} parts per 100,000.
Organic Ammonia . . . . .	0'003	
O <sub>2</sub> absorbed from Permanganate . . . . .	0'060	
(in two hours at 80° F.)		
Total Solid Matters . . . . .	32	
(a) Volatile . . . . .	8	
(b) Fixed . . . . .	24	
(c) Appearance on ignition . . . . .	no charring, etc.	
Total Hardness . . . . .	24	
(a) Temporary . . . . .	19	
(b) Permanent . . . . .	5	
Chlorine . . . . .	2'5	} grains per gallon.
Equivalent to Common Salt . . . . .	4'1	
N as Nitrates and Nitrites . . . . .	0'3	
Poisonous Metals . . . . .	nil	
Microscopical Examination of Sediment. . . . .	—	

*Note.*—The solid constituents of such waters must naturally vary materially in different samples.

*Sample VII.*

## A TYPICAL CLEAR RIVER WATER.

(Derived from deep springs, direct rainfall, and surface drainage.)

*Physical Characters.*—No marked colour. Fairly well aerated. Pleasant taste. No odour. Clear and no sediment—or a very slight one.

Reaction . . . . . Faintly alkaline.

Saline Ammonia . . . .	0'004	} parts per 100,000.
Organic Ammonia . . . .	0'007	
O <sub>2</sub> absorbed from Permanganate . (in two hours at 80° F.)	0'075	
Total Solid Matters . . . .	25	
(a) Volatile . . . .	8	
(b) Fixed . . . .	17	
(c) Appearance on ignition . .	{ extremely faint, discoloration.	
Total Hardness . . . .	21'5	
(a) Temporary . . . .	13	
(b) Permanent . . . .	8'5	
Chlorine . . . .	1'7	} grains per gallon.
Equivalent to Common Salt . .	2'8	
N as Nitrates and Nitrites . .	0'2	
Poisonous Metals . . . .	nil	

Microscopical Examination of Sediment.—A trace of mineral or vegetable debris may be present.

The composition of river water will always of course vary with the following circumstances:—

1. The nature of the country through which the river courses, and which it therefore drains, *i.e.*, whether this be cultivated and manured, or wild; whether there be much or little vegetation, and whether it be thickly or sparsely populated.

2. The amount of pollution by sewage, waste products of manufactories, the drainage of manured land and cemeteries.

3. The nature of the bed of the river, and of the strata through which the springs (which feed the river) rise.

4. The rapidity and smoothness of flow—*i.e.*, the more rapid and interrupted this is, the greater the powers of the river in the direction of self-purification.



*Sample VIII.*

## A DEEP WELL-WATER FROM THE CHALK.

*Physical Characters.*—No marked colour. Pleasant taste. No odour.

Well aerated. Clear. No sediment.

Reaction . . . . .	Alkaline.	
Saline Ammonia . . . . .	0'008	} parts per 100,000.
Organic Ammonia . . . . .	0'003	
O <sub>2</sub> absorbed from Permanganate . . . . .	0'075	
(in two hours at 80° F.)		
Total Solid Matters . . . . .	52	} parts per 100,000.
(a) Volatile . . . . .	10	
(b) Fixed . . . . .	42	
(c) Appearance on ignition . . . . .	no charring, etc.	
Total hardness . . . . .	34	} grains per gallon.
(a) Temporary . . . . .	24	
(b) Permanent . . . . .	10	
Chlorine . . . . .	4'5	
Equivalent to Common Salt . . . . .	7'4	
N as Nitrates and Nitrites . . . . .	0'4	
Poisonous Metals . . . . .	nil	

Microscopical Examination of Sediment. —

*Note.*—The total solids may reach as high as 250 parts per 100,000 in some of these waters, but this amount is very rare.

## SEA-WATER.

The Rivers' Pollution Commissioners found that sea-water contained approximately:—

	PARTS PER 100,000.
Free Ammonia . . . . .	0'006
Chlorine . . . . .	1975'6
Total Solids . . . . .	3898'7

A specimen collected by the late Dr. Tidy during high water at Margate gave :—

Chlorine . . . . .	1770.5
Total Solids . . . . .	3343.0
Lime . . . . .	35.1
Magnesia . . . . .	205.6
Silica . . . . .	0.4
Hardness . . . . .	564.0

#### SCHEME FOR EFFECTING AN ANALYSIS IN THE QUICKEST AND MOST CONVENIENT MANNER.

(As by this scheme more than one process is going on at the same time, care must be taken that all due attention is given to each ; although a little confusion may be experienced at first, yet, after a little practice, the plan will be found as practical as it is expedient).

1. Remove 100 c.c. of the water from the sample bottle, and start the evaporation over a water-bath for "the total solids in solution."

2. Remove 250 c.c. and commence the estimation of "the oxidisable organic matter."

3. Remove 500 c.c. from the bottle, and start the distillation for the estimation of "saline" and "organic" ammonia.

Then start the evaporation of a litre of water, acidified by hydrochloric acid, to 200 c.c. for the purpose of testing for poisonous metals, etc.

4. Decant about 500 c.c. of the water into a clean conical glass ; cover this, place aside, and allow the bulk of any suspended matter present to settle.

5. Estimate the hardness, "total," "temporary," and "permanent."

6. Make the "quantitative estimation of chlorine."

7. Note the physical characters in the two-foot tube, etc., together with the reaction of the water.

8. Test for poisonous metals, and estimate quantitatively if any one is present.

9. Test for nitrites, sulphates, and phosphates, and estimate quantitatively if necessary.
10. Make a microscopic examination of any sediment or suspended matter.
11. Prepare the water for the quantitative estimation of "nitrogen as nitrates and nitrites" by the wet copper-zinc couple process, set aside, and complete the process on the following morning.

#### SCHEME FOR EXAMINATION PURPOSES.

(In the examination for Public Health Degrees, the time is limited, and the subjoined tests are considered to suffice in detecting whether the candidate has a good knowledge of the subject of water analysis or not. He is expected to hand in an abstract of each process by which he arrives at his results, in addition to the results themselves).

1. Start the quantitative estimation of free and albuminoid ammonia\* and then proceed to determine the following points:—
2. The physical characters, *i.e.*, colour, clearness, aeration, reaction, taste, smell, sediment (if any).
3. The quantitative estimation of chlorine.
4. The total hardness.
5. The qualitative examination for poisonous metals and calcium salts.
6. The qualitative examination for nitrates, nitrites, sulphates, and phosphates.

7. The macroscopic appearances of any sediment present. (If the necessary apparatus and reagents are provided, a quantitative estimation of "nitrogen in nitrates and nitrites," and of the "oxidisable organic matter," may be required. The former estimation should be performed by either the

\* Step 4 will be completed by the time the free ammonia is ready for Nesslerisation.

“picric acid” or “indigo” process, and started immediately after Wanklyn’s process is commenced).

#### AN ANALYTICAL FORM.

**If a solid residue:**—(1) Dissolve as much as possible by boiling in distilled water; (2) dissolve any residue by boiling in dilute hydrochloric acid; (3) dissolve any residue still remaining by warming with strong hydrochloric acid and nitric acid, mixed in the proportion of 2 parts to 1.

To some of the solution thus obtained:—

A. Add a few drops of **hydrochloric acid**, until the maximum precipitate—if any—is created.

(a) *A white precipitate* = silver, mercury (-ous salt), or lead; *add ammonia*, if the precipitate dissolves = silver; if it blackens = mercury; if unchanged = lead.

Add a solution of *potassium chromate* to some of the original liquid; a reddish-brown precipitate = silver; a red precipitate = mercury; a yellow precipitate = lead.

B. If no precipitate add **strong sulphuretted hydrogen water**.

(a) *A yellow precipitate* = arsenic, tin (-ic salt), or cadmium. If arsenic or tin (-ic), the precipitate is soluble in ammonium sulphide and in potassic hydrate solution. Distinguish by Reinsch’s test. If cadmium the precipitate is more of an orange colour, and it is insoluble in ammonium sulphide and potassic hydrate solution.

(b) *An orange-red precipitate* = antimony.

Add *potassic hydrate* solution to original liquid, and a white precipitate forms, soluble in excess.

(c) *A brown precipitate* = bismuth, copper, or tin (-ous).

If bismuth or copper, the precipitate is insoluble in ammonia and potassic hydrate solution.

If tin (-ous), the precipitate is soluble in ammonia and potassic hydrate solution.

Copper will give a bronze colour and precipitate with potassium ferrocyanide.

(d) *An orange or black precipitate* = mercury (-ic, salt).

A solution of potassium iodide will give a scarlet red colour to the original liquid.

C. To a fresh portion of the solution add a solution of the **chloride of ammonium, and ammonia.**

(a) *A brownish black precipitate* = iron; ferrocyanide of potassium will yield a blue colour; ferricyanide of potassium a red with ferric and blue with ferrous salts.

(b) *A greenish-white precipitate* = aluminium.

(c) *A dark green precipitate* = chromium.

D. If no precipitate, further add some **ammonium sulphide** solution.

(a) *A white precipitate* = zinc.

(b) *A salmon-colour precipitate* = manganese.

(c) *A black precipitate* = nickel or cobalt.

If nickel, ammonia yields a blue; if cobalt, ammonia yields a green.

E. If no precipitate, add **ammonium carbonate** solution.

(a) *A white precipitate* = barium, strontium, or calcium.

If barium—burns with a green flame; if strontium and calcium—burns with a red flame; potassium chromate creates a yellow colour with strontium and none with calcium.

F. If no precipitate is obtained by this stage, only magnesium, ammonium, potassium, sodium, and lithium can be present.

Add **ammonium chloride, ammonia and sodium phosphate.**

(a) *A white precipitate* = magnesium.

If *no precipitate*, but ammonia is evolved on boiling the original solution with sodic hydrate = ammonium.



If potassium, the original solution on platinum burns with a lilac flame; if sodium, it burns with a yellow flame; if lithium, a crimson.

Potassium will furnish a yellow precipitate with platinum chloride solution.

**For acids.** To original solution:—

A. Add **barium chloride solution** (or if silver, mercury, or lead be present, use barium nitrate).

(a) *A white precipitate* = sulphates, carbonates, phosphates, oxalates, or borates.

Add *hydrochloric acid*.

If sulphates, the precipitate is insoluble even on boiling; if carbonates the precipitate is soluble (also in acetic acid) with *effervescence*; if phosphates or borates the precipitate is soluble (also in acetic acid); if oxalates the precipitate is soluble (but not in acetic acid).

B. If no precipitate, to some of the original solution add **silver nitrate**.

(a) *A white precipitate* = chlorides or cyanides; add *nitric acid*.

If chlorides the precipitate is insoluble (and it *fuses* on heating); if cyanide the precipitate is soluble on boiling.

(b) *A pale yellow precipitate* = bromides or iodides; add a drop of strong *sulphuric acid*.

If bromides, starch paper is coloured orange; if iodides, starch paper is coloured blue.

C. **Test especially for:**—Nitrates; sulphides—heat with acid, and sulphuretted hydrogen evolved; acetates—heat with dilute sulphuric acid (vinegar smell) and if ferric chloride solution is added to a neutral acetate the liquid gets deep red (ferric acetate), and on the addition of hydrochloric acid turns yellow; tartrates—heat with strong sulphuric acid, blacken *immediately*, and burnt sugar odour and sulphurous acid smell evolved; citrates—treated simi-

larly to tartrates, blacken *slowly* and pungent irritating fumes are given off.

*Salicylic acid*.—Add a solution of the perchloride of iron, and a deep purple colour is formed.

*Boracic acid* and *borates*.—Evaporate the liquid, after rendering alkaline, to a solid residue, ignite, and then add a few drops of fuming hydrochloric acid to the ash and evaporate over the water-bath. While this is proceeding, direct a Bunsen flame across the top of the evaporating dish, and it will acquire a yellowish green tinge, but if in minute traces the border of the flame alone shows a greenish tint. Or add about a drachm of spirit of wine to the ash and acid, warm together, and then burn the spirit, and observe a bright green flame. For a still more delicate method *vide* Milk Analysis.

#### SEWAGE EFFLUENTS.

The analysis of a sewage effluent proceeds upon similar lines to those of a water analysis, but in these cases it is necessary to dilute the sewage matter to a considerable extent before commencing the estimation. So far as the solids are concerned, these may be estimated from the original effluent, but for the calculation of the two ammonias and oxidisable organic matter, 10 c.c. of the effluent should be made up to the litre with ammonia-free distilled water, and the results obtained when multiplied by 100 will represent the amounts in the original effluent. In Nesslerising the ammonias the contents of the Nessler glasses after treatment with the reagent may be all mixed together in a beaker, and the colour of 50 c.c. matched; thus supposing 200 c.c. of the distillate were collected before all the free ammonia had come over, then the ammonia estimated in 50 c.c. after mixing the whole 200 c.c. together, must be multiplied by 4. The nitrates may be estimated from the original effluent by the picric-acid method.

The composition of sewage from the same district varies very greatly from time to time, and this is true, though to a far less extent, of the effluent. The effluent, moreover, varies according to the method of treatment, but the following mean results obtained by the writer of many analyses may serve to give the reader a general idea of the composition of sewage and sewage-effluents:—

<i>Sewage as it leaves the outfall sewer.</i> (Parts per 100,000.)				<i>Effluent after the sewage has been chemically treated, and the supernatant liquid from the tanks has been let upon the land.</i> (Parts per 100,000.)	
Free and saline ammonia	...	7.5	...	1.5	
Organic ammonia	...	1.8	...	0.5	
O <sub>2</sub> absorbed in 2 hours at 80°F.	12.6	...	...	4.2	
Nitrogen as nitrates and nitrites	0.005	...	...	1.2	
Chlorine	...	11.1	...	10.0	
Suspended matter	...	45	...	3.5	
Solids in solution	...	98	...	87	
(a) volatile	...	52	...	35	
(b) non-volatile	...	46	...	52	
Alkalinity	...	...=85 parts Na <sub>2</sub> CO <sub>3</sub>		=35 parts Na <sub>2</sub> CO <sub>3</sub>	

#### ALKALIMETRY AND ACIDIMETRY.

For the purposes of estimating the degree of alkalinity or acidity of any fluid, it is convenient to use standard solutions based upon the atomic or molecular weights of the different reagents, or made up in such a way that equal volumes of the solutions are made chemically equivalent to each other.

Such solutions are "normal" when they contain in one litre at 60.8 deg. F., chemically equivalent weights of the active reagents weighed in grammes, hydrogen being taken as the unit. The normal solution of hydrochloric acid must therefore contain the molecular weight of the acid, *i.e.*, 36.37 in grammes per litre, since HCl is a univalent substance. The normal solution of sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) must con-

tain the molecular weight (106) divided by 2 = 53 in grammes per litre, for the molecule of monobasic HCl can neutralise only half a molecule of  $\text{Na}_2\text{CO}_3$ .

"Seminormal" and "decinormal" solutions are obviously those made up to  $\frac{1}{2}$  and  $\frac{1}{10}$  respectively of the strength of the "normal" solutions.

Each cubic centimetre of a normal solution of HCl will therefore contain  $\frac{1}{1000}$  of the molecular weight of the acid in grammes (*i.e.*, 0.03637 gramme), and a decinormal solution contains  $\frac{1}{10000}$  (*i.e.*, 0.003637 gramme); each cubic centimetre of the normal acid will exactly neutralise 0.053 gramme of sodium carbonate in a normal solution, and 0.0053 gramme in a decinormal solution.

Measured quantities of normal or decinormal acids, then, should exactly neutralise similar quantities of the normal or decinormal alkalies, and if, on titration, they are not found to quite correspond, the difference must be ascertained and a simple calculation made in order to get their relative values.

In estimating alkalinity the decinormal solution of hydrochloric acid may conveniently be employed; and for acidity the decinormal solution of sodium carbonate. In each case one of these standard solutions would have to be run in in measured quantities, until the neutral stage is exactly reached, as indicated by a suitable reagent which is added to the solution.

Methyl-orange (about 1 gramme to the litre) is a good "indicator" where water is being tested. This substance has the property of yielding a beautiful scarlet colour in the presence of acidity; but its solution, which is of a bright orange colour, must not be employed where organic acids are concerned, or where nitrites are present. In the latter case phenolphthalein may sometimes be substituted. Litmus should not be used as an "indicator," for the free  $\text{CO}_2$  so commonly present in water considerably masks its indications. A solu-

tion of cochineal is almost free from this drawback, and is a useful indicator. It is prepared by digesting the dried and powdered cochineal in warm water, to which a little alcohol has been added, and then filtering; the solution has a yellow or yellowish-red colour, which is turned violet-red by alkalis, and the original colour is restored by mineral acids.

*Example.*—It is desired to estimate the alkalinity of a water sample. A few drops of the methyl-orange “indicator” are run into a measured quantity—say 100 c.c.—of the water in a white porcelain dish. The decinormal solution of HCl is then dropped in from a graduated burette until evidence of a scarlet tint appears, denoting all alkalinity to be neutralised. It took 6 c.c. of the decinormal acid to effect neutrality, therefore the alkalinity is equivalent to 6 c.c. of this acid solution. But 6 c.c. of the decinormal HCl is equivalent to a similar amount of decinormal sodium carbonate solution, therefore the alkalinity is equivalent to 6 c.c. of the decinormal sodium carbonate solution.

But 1 litre of the normal solution contains 53 grammes of sodium carbonate; therefore 1 litre of the decinormal solution contains 5.3 grammes, 1 c.c. of this contains 0.0053 gramme, and 6 c.c. contain 0.0318 gramme of sodium carbonate.

Therefore the alkalinity of 100 c.c. of the solution is equivalent to 0.0318 gramme of sodium carbonate.

In estimating acidity, the “indicator” would be added, and the sodium carbonate decinormal solution run in until the scarlet colour is just discharged, when the calculation is made as above.

To prepare the normal HCl it is necessary to take about 181 grammes of liquid acid of the S.G. 1.10, and dilute to a litre with water, then titrate the exact strength with normal sodium carbonate.



## PART II.

### SOIL EXAMINATION.

#### THE ANALYSIS OF SOILS.

THE Sanitarian will not often find it necessary to make a chemical analysis of soil, and where he does it must be very rarely required that the analysis should proceed beyond a qualitative estimation of those various constituents which may, on account of their ready solubility, gain access to drinking water; if, moreover, the subject is looked upon from an agricultural point of view, the easily soluble constituents of the soil will alone concern the analyst, since these alone are taken up by plant life. It will be necessary, then, to include in the analysis those substances which are soluble in a weak acid medium, since such acidity is sometimes naturally supplied in the form of vegetable and peaty acids, carbonic acid, etc.

All soils contain, though in different proportions, the chief mineral constituents which are found in the ash of the plants which grow upon them; and an analysis of such ash will afford a rough and ready clue to the constitution of the soil.

The same value is not attached to an analysis of the aqueous extract of soils as was formerly the case, for it is now well understood that such does not contain the same proportions of the soluble constituents as existed in the original soil. This is accounted for by the fact that soil will yield up readily to water the substances in regard to which its powers of absorption have been satisfied, while it will more or less strongly retain other soluble ingredients.

Whereas the power of absorbing and retaining moisture is a consideration of the first importance from a health view—exercising as it does an important influence upon the health of whole communities—yet it is of little practical value to perform any tests in this direction upon small quantities of soil, which are collected and brought to a laboratory. The powers of retaining moisture are so largely dependent upon the characters of the subsoil, etc., that the most reliable and valuable information is always obtained by observations of the soil *in situ*. The depth of the ground water and its degree of fluctuation should be always ascertained.

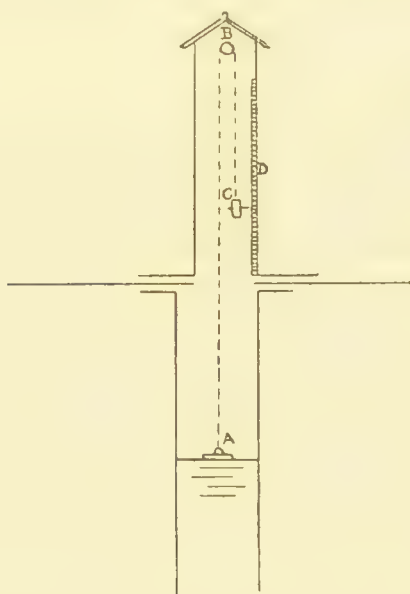


FIG. 34.—Arrangement for registering the height of the ground-water:—A. A float; B. A pulley; C. An index; and D. A graduated scale.

The method of testing the capacity which the soil possesses of holding water is obvious:—The dried soil is weighed in a cylinder, and then saturated with water; the water is allowed to drain off through very fine muslin until no more drops fall,

and the soil is then reweighed ; the difference in weight represents the weight of water it is capable of holding.

In collecting samples, it must be borne in mind that the characters of the soil may vary within small areas and at different depths—so that many samples must be collected and analysed before one can speak with anything approaching accuracy, of the constituents of a soil of a large area. The depth of the surface soil varies considerably in different localities. In uncultivated grounds the soil generally occupies only a few inches in depth on the surface, and in cultivated grounds its depth is generally the same as that to which the implements used in cultivation have penetrated. Soil is composed of certain mixtures or combinations of the following substances:—

The earths—silica, alumina, lime and magnesia ; the alkalis—soda, potassa, and ammonia ; the acids—sulphuric, hydrochloric, carbonic, nitric, phosphoric, silicic, and humic ; oxide of iron, and small portions of other metallic oxides ; a considerable proportion of moisture ; and several gases. Besides these every soil contains a large amount of vegetable and animal matter which is either partially or wholly decomposed, and which is ultimately converted into water, carbonic acid and nitric acid.

The stratum which lies immediately under the soil is called the *subsoil*, into the composition of which comparatively little organic matter enters ; sometimes this subsoil is porous sand or gravel ; sometimes light and loamy and closely similar to the super-imposed soil ; sometimes stiff (clayey) and more or less impervious to water.

*The Analysis.*—The sample having been collected, the coarser stones should be removed, and all lumps broken up so far as possible with a hard wooden pestle.

Special apparatus has been devised, both for thoroughly crushing and also for washing and separating, by sieves of

different sized meshes, the various coarse constituents seriatim: thus the small rock fragments (of over 10 m.m. in diameter) are first separated, then the small stones or gravelly sand (of from 2—5 m.m.), next the coarse sand (of 1—2 m.m.), the fine sand (of 0.4—1 m.m.), and the clayey sand (below 0.4 m.m.), until finally the finest clayey portions only remain.

To constitute pure clay the particles should not exceed 0.01 m.m., and the material should be previously treated with sufficient hydrochloric acid to dissolve out any carbonates; the washed soil should then be heated with 3 per cent. ammonia to dissolve humus, washed, dried, ignited, and weighed.

The amount of sand in clay is usually estimated as follows:—

Heat a weighed quantity of the dried material with sulphuric acid; then boil with water, collect the insoluble matter on a tarred filter, dry, and weigh; remove and boil an aliquot part of this insoluble matter with a strong solution of sodium carbonate, and weigh the insoluble residue as sand.

But essentially the analysis is of importance in detecting the substances which a water will extract and hold in solution, and hence this is made the chief feature of the analysis here. Schulze's method (which is recommended by Fresenius) for obtaining the aqueous extract of a soil is as follows:—The necks of several middle-sized funnels are closed with small filters of strong filter paper; these are moistened, and the paper pressed close to the sides of the funnel; the air-dried soil is then introduced in small lumps ranging in size from a pea to a walnut (but not pulverised or even crushed), until the funnels are filled to about two-thirds. Distilled water is now poured in in quantity sufficient to cover the soil. If the first portion of the filtrate is turbid it must be poured back into the funnel, and the filtration allowed to proceed quietly; the funnels are again filled with water, and this process of extraction is continued until the filtrates weigh

twice or three times as much as the soil used. The several filtrates are next mixed in one vessel, and a portion of the washed soil is kept. The aqueous solution is divided into a larger and a smaller part, and the larger is evaporated to a small bulk. A part of this concentrated solution is then tested for organic matter and chlorine, and the remainder (which generally contains a little precipitate) is evaporated to dryness and cautiously ignited, so that the organic matter may be slowly but completely burnt off; some of the ash thus obtained is tested in the dry for manganese, by fusing it with two or three parts of sodium carbonate upon a platinum wire; the bead of manganate of soda thus formed appears as a transparent green while hot, and an opaque bluish-green when cold. The remainder of the ash is dissolved in hydrochloric acid, when any effervescence indicates carbonates. The whole is then evaporated to dryness, then moistened with hydrochloric acid, water added, and the mixture warmed and filtered. The filtrate is next tested for sulphuric acid, phosphoric acid, iron, and (if required) for magnesia, potassa, soda, and lithia. The residue generally contains a little carbon, a little clay, and also silicic acid, and the silicic acid may be tested for by washing the residue, boiling it with caustic soda, filtering, saturating with hydrochloric acid, evaporating to dryness, and finally taking up the residue with water—when the silicic acid will be left behind.

Alumina was never found by Schulze in the aqueous extract.

In most soils the phosphoric acid exists as a basic ferric phosphate, and hence the great insolubility of soil phosphates.

The smaller part into which the non-concentrated aqueous solution was divided is finally tested for nitric and nitrous acids and ammonia.

The portion of the soil insoluble in water averages about 90 per cent. of the total, and the analysis then proceeds to deal with this in the following manner:—

About 50 grammes of the washed soil are heated for several



hours with hydrochloric acid (of medium strength) upon a water-bath, and then filtered. The filtrate, which is often yellow from ferric chloride, contains the substances in the soil which are soluble in an acid medium; and it is accordingly tested for iron, manganese, copper, alumina, lime, fluorine, magnesia, potassa, soda and lithia, silicic, phosphoric, sulphuric, carbonic, and even arsenic acids.

The phosphoric acid in soils is estimated by Gasparin in the following way:—20 grammes of the finely powdered and sifted earth are treated with sulphuric acid (1 : 5) in a porcelain dish until effervescence ceases; 80 c.c. of aqua regia (1 nitric acid : 3 hydrochloric acid) are then added, and the mixture heated on the water-bath till the liquid becomes syrupy; it is then diluted with cold distilled water and washed on to a filter with hot water; the filtrate is next precipitated with ammonia, collected and dried. The dry precipitate is heated in a platinum crucible to redness, digested with cold dilute nitric acid (1 : 40) and filtered; the filtrate (containing the phosphoric acid) is concentrated on the water-bath, precipitated with molybdic acid, and the phosphoric acid determined as magnesium pyrophosphate.

As the solvents which act naturally on the soil, however, are far weaker than the hydrochloric acid here employed, it will be more exact if we examine those substances which are soluble in carbonic acid water—as by saturating distilled water with carbonic acid and allowing this to act upon the soil for several days in a closed flask which should be well shaken from time to time. Water containing both carbonic acid and ammonium chloride (about 0.05 per cent.), should also be allowed in a similar manner to act upon the soil, and the substances *then* taken up should be examined.

Probably the best solvent for extracting “available,” as distinguished from “total,” mineral constituents of plant food from soil, is a 1 per cent. solution of citric acid (Bernard Dyer).

No practical hygienic purpose will be served by analysing that part (which is always the greater) of the soil which is insoluble even in an acid medium—it then becomes chiefly a matter of finely crushing, and estimating silicates.

An examination for the *peaty acids* may be made thus :—Some of the washed soil is dried and sifted, to separate any straw, roots, and stones ; what passes through the fine sieve is digested for several hours at 80 to 90 deg., with a solution of carbonate of soda, and filtered ; the filtrate is then slightly acidified with hydrochloric acid, and if brown flakes separate, these consist of the peaty acids, *i.e.*, ulmic, humic, or geic. The more ulmic acid is present the lighter is the shade of brown ; a dark shade indicates a preponderance of humic or geic acids.

Collect these flakes upon a weighed filter, wash until the water begins to be coloured, dry, and weigh. Burn the dry mass, deduct the weight of the ash (after subtracting the filter ash) from that of the dry mass, and enter the difference as “acids of humus.”

It is occasionally desirable to know whether the soil has been recently polluted with excremental matter ; the aqueous extract can then be tested for oxidised nitrogen, chlorine and organic matter, and the amounts found compared with those procured from similar soil in the neighbourhood. The total nitrogen would be best determined by Kjeldahl's process.

Kjeldahl's method is easy of execution and accurate. The organic substance is exposed in a dry finely pulverised state to the action of concentrated sulphuric acid, raised to the boiling point, and after a while, when the contents of the flask have become clear and practically colourless, small quantities of the permanganate of potash, in a finely powdered state, are added, until a green or pink colour becomes established—in order to ensure complete oxidation ; the nitrogen thus becomes converted to ammoniac sulphate. Let cool,

dilute a little with water, add excess of caustic soda, and distil off the ammonia into seminormal hydrochloric acid and titrate it with seminormal soda solution. Cochineal is recommended as the indicator. A good detailed account of the process may be seen in Lehmann's "Methods of Practical Hygiene," pages 390-394.

The estimation of carbonic acid, etc., in the ground air, is conducted upon lines given in Air Analysis (see Part III).

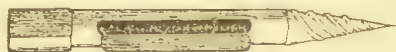


FIG. 35.—Fraenkel's borer, for taking samples of soil from any depth.

An ordinary microscopical examination of the soil may sometimes be made with advantage. The microbes in soil, their characters, and what they effect, are as yet but partially understood, and they afford an interesting subject for further research; they are very numerous, and vary both in their morphological characters and in their actions. The class which attacks organic matter with the production of oxidised nitrogen and carbonic acid are relatively of the greatest importance; they mainly occupy the upper 3 feet of soil, and exist in greatest numbers in the warmer open porous soils rich in organic matter, and the presence in the soil of some substances such as calcium and magnesium carbonates is a necessity for their continuous action. In addition to these so-called "nitrifying organisms," there are others which reduce nitrates to ammonia, and still others that will fully oxidise ammonia in the presence of air, but will reduce nitrates to ammonia in the temporary exclusion of air.

The results, obtained by the writer, of a mineral analysis of a few common soils are here appended; but it must be understood that those soils which are called by the same name may vary considerably in the nature and amounts of their less

characteristic constituents. The main purpose of the following analyses is to afford an *approximate* idea only of the amount of the various substances which enter into the composition of the more common soils.

CLAY (*Stourbridge*).

Silica	...	...	...	...	68
Alumina	...	...	...	...	15
Organic matter	...	...	...	...	4
Iron (oxide)	...	...	...	...	3
Lime	...	...	...	...	1.5
(carbonate 1.4)					
(sulphate 0.1)					
Magnesia, etc.	...	...	...	} traces	0.5
Phosphoric acid	...	...	...		
Water	...	...	...	...	8
					<hr/>
					100.0

CALCAREOUS (*Sussex*).

Lime	...	...	...	...	90
(carbonate 89.5)					
(sulphate 0.35)					
(phosphate 0.15)					
Magnesia (carbonate)	...	...	...	...	0.5
Oxide of iron and alumina	...	...	...	...	2.5
Silica	...	...	...	...	0.55
Organic matter	...	...	...	...	3
Water	...	...	...	...	3.45
					<hr/>
					100.00

PEATY (*Devonshire*).

Organic matter	...	...	...	90.5
Silica	...	...	...	7.5
Alumina	...	...	...	0.74
Lime	...	...	...	0.5
Sulphuric acid	...	...	...	0.2
Oxide of iron	...	...	...	0.46
Magnesia	...	...	...	0.05
Phosphoric acid	...	...	...	0.02
Soda and potash	...	...	...	0.03
				<hr/>
				100.00

GARDEN VEGETABLE MOULD.

Silica	...	...	...	49.25
Organic matter	...	...	...	13.5
Oxide of iron	...	...	...	9.25
Carbonic acid	...	...	...	7.12
Water	...	...	...	6.9
Lime	...	...	...	5.13
Alumina	...	...	...	2.74
Soda and potash	...	...	...	2.5
Chlorine	...	...	...	1.5
Sulphuric acid	...	...	...	1.3
Oxide of manganese	...	...	...	0.25
Phosphoric acid	...	...	...	0.4
Magnesia	...	...	...	0.16
				<hr/>
				100.00

If it is desired to classify a soil, it can readily be effected in the following manner:—

A weighed quantity should be mixed with water, shaken thoroughly, and then placed at rest; in a few minutes the



sand will settle, while the fine lighter particles of clay will remain suspended. If the water and clay is decanted and set aside, after a time the water becomes clear, and the clay can be collected, dried, and weighed.

Any soil which is not so rich in vegetable matter as to constitute a "peaty" one, and which contains—when thus treated—not over

10 per cent. of clay = a "sandy soil."

10-40 per cent. of clay = a "sandy loam."

40-70 per cent. of clay = a "loamy soil."

70-85 per cent. of clay = a "clay loam."

85-90 per cent. of clay = a "strong clay soil."

A soil containing no sand at all = a "pure agricultural clay"—which is essentially a silicate of alumina, mixed with small quantities of organic matter, lime, magnesia, and oxide of iron. The different varieties are mainly due to the varying amounts of these latter substances.

If there is more than five per cent. of calcium carbonate, the soil is called "a marl"; and if there is more than twenty per cent., "calcareous."

"Peaty" soils generally contain from sixty to eighty per cent. by weight of organic matter; "rich cultivated soils" from about five to twenty per cent.; and "stiff clayey" ones from two to ten per cent.

By means of vegetation, and owing to the fixation of free nitrogen by soil micro-organisms and plants, even a sandy soil may in time become converted into a productive soil.

Strong clays absorb and retain nearly three times as much water as sandy soils, while peaty ones absorb a still larger proportion; and the same remarks broadly apply to the relative readiness with which water is lost by evaporation from these soils.

In 100 parts of soil dried in the air, Krockner found that clayey soils, before manuring, yielded 0.1 to 0.45 of ammonia;

loamy soils, 0.13 of ammonia ; sandy soils (never cultivated), about 0.05 of ammonia ; marls, 0.004 to 0.09 of ammonia.

As would be inferred, the soil of graveyards above the burial level does not materially differ, as regards amount of organic matter and its products, from similar soil (unmanured) elsewhere ; but that taken on the level of the coffins and from a short distance beneath, is relatively richer in organic matter. Such soil is found to be somewhat richer in bacteria than other unmanured soils, and more especially is this the case with that lying around the top of the coffins (Reiners, Fraenkel, Young).

The various **manures** with which the soils under cultivation are dressed of course effect considerable changes in the constitution of the original soil, besides yielding abundance of soluble matter to the water which comes in contact with them.

The commoner manures are :—

Farmyard and animal excrement, and “Guano.”

Bone dust, and other phosphatic manures (calcium phosphate, etc.).

Vegetable manures—sawdust, soot, charcoal, peat, and seaweed.

Ammonia salts—especially the sulphate.

Sodium salts—especially the nitrate.

Potassium salts—especially the chloride, nitrate, and phosphate.

Gypsum.

## PART III.

## AIR ANALYSIS.

## CHAPTER I.

THE NORMAL AND ABNORMAL CONSTITUENTS OF AIR.  
EUDIOMETRY.

A HYGIENIC analysis of atmospheric air aims at detecting the injurious gases and vapours which it may contain, together with the nature and amount of the suspended matter present.

Atmospheric air, the elastic fluid which we breathe, consists, almost *in toto*, of a mixture of the gases—nitrogen and oxygen; it is possessed of gravity, fluidity, and of the power of becoming rarified and condensed; and since all gases have now been resolved, by reduction of temperature and increase of pressure, into liquids, they differ only from those bodies in their degree of elasticity and in the aeriform condition in which they exist. It is advantageous to recognise this at the very outset, for a knowledge of the fact will serve to make clear a good deal in the methods of treating gases for analytical purposes.

Broadly the atmospheric air consists of:—

Oxygen	...	...	...	20.96
Nitrogen	...	...	...	79
Carbonic acid	...	...	...	0.04
				<hr/>
				100.00
				<hr/>

After this air has been respired, the carbonic acid is increased to the extent of about 4 per cent., and there is a corresponding diminution in the oxygen, *i.e.*, expired air contains about:—

Oxygen	...	...	...	...	16.96
Nitrogen	...	...	...	...	79
Carbonic acid	...	...	...	...	4.04
					<hr/>
					100.00
					<hr/>

and in addition to this change in its composition, it has gained "organic matter" and is saturated with aqueous vapour.

An analysis of ordinary air will detect, however, in addition to oxygen, nitrogen, and carbonic acid, the presence, in traces only, of suspended matter, and also of other gases. The average composition of country and town air is as follows:—

## COUNTRY AIR.

Nitrogen	. . . . .	79.00
Oxygen	. . . . .	20.90—20.94
Carbonic acid.	. . . . .	0.03—0.04
Aqueous vapour	. . . . .	{ variable (from 0.4 to 1.6)
Ammonia	. . . . .	traces
Oxidised nitrogen	. . . . .	"
Ozone	. . . . .	"
Marsh gas	. . . . .	"
Common salt and other mineral substances	. . . . .	"
Organic matter—living or dead (including micro-organisms)	. . . . .	"

## TOWN AIR.

(Reaction—faintly acid)

Nitrogen	. . . . .	79.00
Oxygen	. . . . .	20.86—20.92 (average)
Carbonic acid	. . . . .	{ 0.04—0.05 (average 0.07 during fog)
Aqueous vapour	. . . . .	variable

Sulphurous acid, sulphuric acid, and sulphurated hydrogen . . . . .	marked traces
Ammonia . . . . .	traces
Oxidised nitrogen . . . . .	„
Marsh gas . . . . .	„
Common salt and other mineral substances . . . . .	„
Organic matter—more than in country air (including micro-organisms) . . . . .	„

It will thus be seen, that, with the exception of oxygen and nitrogen, everything else which may be found in the general atmosphere exists only in the very smallest proportions, and as it is these latter constituents which chiefly concern the sanitarian, it will be gathered that any processes adopted for their estimation must be delicate, and carefully performed, and that large bulks of air should be employed.

There is not, under ordinary circumstances, much to be gained by a sanitary examination of the general atmosphere, except it be with regard to its moisture, or during the occasion of city fog. The chief scope for such work lies in the unhealthy air of crowded rooms (as to its organic matter, etc.), of manufactories (as to the presence of poisonous gases from the chemicals, etc., employed), of mines (for poisonous and explosive gases), of cellars (for “ground air,” “sewer gas,” etc.).

What then are the gaseous and volatile substances which may, under favourable conditions, gain access to the air, and what is their source? It is obvious, with regard to the latter question, that their source must be either from the vital processes of organic life (respiration and transpiration), the natural decomposition of organic matter, the combustion of various materials, or from the products, etc., of trade processes.

They may be thus classified:—

*Carbon and compounds of carbon*:—Carbon (from incomplete combustion).

Carbonic oxide (from combustion, iron and copper stoves).



Carbonic acid (from combustion, respiration, organic decomposition, chemical works, soda water manufactories, brewing, etc.).

Carburetted hydrogen (from combustion, organic decomposition).

Carbo-ammoniacal substances (from sewers, etc.).

*Compounds of sulphur*:—Sulphurous acid (from combustion—and varying in amount with the pyrites in the fuel, bleaching works, copper smelting, volcanic action).

Sulphuric acid (from combustion).

Sulphuretted hydrogen (from combustion, putrefaction, chemical works, etc.).

Ammonium sulphide (from combustion, putrefaction).

Carbon bisulphide (from combustion, vulcanised india-rubber works, etc.).

*Chlorine and compounds of chlorine*:—Chlorine (from bleaching works).

Hydrochloric acid (from alkali works, etc.).

<i>Compounds of nitrogen.</i> —	{	Ammonia.	{	From organic decomposition, and from combustion.
		Nitrous acid.		
		Nitric acid.		
		Ammonium sulphide.		
		Ammonium carbonate.		

*Organic vapours*, the composition of which is undetermined (from decay of organic matter, trade processes—such as soap works, glue refiners, bone boiling and burning, tanyards, etc.).

*Metallic fumes* of arsenic (from copper smelting, etc., *vide* arsenic).

"	"	copper (from copper smelting).
"	"	lead (from various trade processes).
"	"	zinc (from brass founding).
"	"	phosphorus (from match making, especially before the red or amorphous phosphorus was employed).

*Moisture* (from combustion, respiration, evaporation, etc.).

The next point is to consider upon what *principles* we shall proceed to learn the nature and proportions of the constituents of any gaseous mixture.

Some chemical substance might be added with which any particular gas has the property of combining, and the amount of such gas which has thus combined may be readily estimated by the increased weight, etc., gained by the substance which was exposed to it; this plan, however, is not very satisfactory, and is not, in consequence, generally employed. The only other practical alternative remaining is to estimate the amount of gas which has thus been absorbed, by ascertaining what loss in *volume* the original mixture has suffered.

The eudiometer (*ευδῖος*, serene, good, and *μέτρον*, measure) is the instrument employed for measuring the volume of a gas, or gaseous mixture. In order to demonstrate the most simple and ready manner of performing eudiometry, the estimation of the amount of oxygen in a sample of air is chosen. It is obvious that the process must be conducted with great care, and that, dealing as we are with a gas, the different volumes must be measured off under exactly the same conditions of temperature and atmospheric pressure, and that—varying as these may from time to time—the process of estimation should not be unduly prolonged.

We have seen that the amount of oxygen in the external atmosphere may be taken to constitute a normal percentage of 20.96. After a careful consideration of the number of investigations which have been made, it seems that it may reach its highest limit of 21 per cent. over large expanses of open country; and that in the most crowded parts of cities it seldom, if ever, falls below 20.75 in the external atmosphere (even in the time of fog), or below 20.65 in the atmosphere of occupied rooms.

In mines, however, the oxygen has been estimated as con-

siderably below 20 per cent. (Angus Smith found 18.27 per cent., and some Continental observers have estimated it even lower). During fog and frost in Manchester, and in the pit of a theatre, the same observer found 20.89 per cent. and 20.74 per cent. respectively.

Having in view the fact that the amount of oxygen diminishes to such a slight extent in atmospheres which are foul and unhealthy, it is clearly not by the estimation of this element that much information of a sanitary nature can most readily be gleaned, and its introduction here is mainly because it serves admirably to illustrate the performance of an eudiometric observation, the principles of which the reader must early become conversant with. It must not be thought, however, that small though the difference is between 20.96 per cent. and 20.75 per cent., *i.e.*, 0.21 per cent., it may be altogether ignored, for the loss in oxygen is made up by some deleterious agent or agents—and these will be present in a million volumes to the extent of 2,100 volumes!

#### THE ESTIMATION OF THE AMOUNT OF OXYGEN IN THE ATMOSPHERE BY EUDIOMETRY.

**Apparatus required.**—Many of the gas-measuring apparatus are cumbersome, costly, and difficult to work, and the writer has found one of the most simple and convenient of these instruments to be Hempel's gas burette. From the accompanying figure it will be seen to consist of two glass tubes supported on flat round stands, and connected together at their lowest points by wide india-rubber tubing; the tube which is seen in fig. 36 to be held up (and which will be subsequently referred to as tube A) is plain, and is continued full bore up to the top, where it generally ends in a slightly trumpet-shaped mouth; the other tube (which will be referred to as tube B) is graduated into cubic centimetres, and narrowed above so as to fit inside of a piece of small india-rubber

tubing, which serves to connect it with the apparatus ("absorption pipette") containing the chemical substances em-

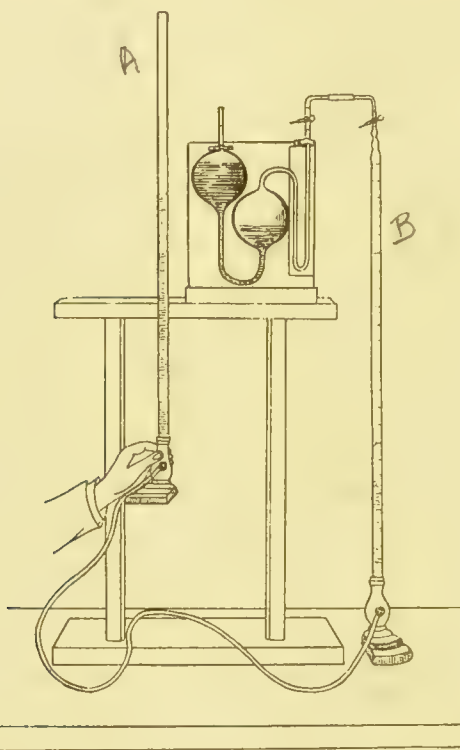


FIG. 36.—Hempel's gas burette and absorption apparatus.

ployed to absorb and remove any constituent of a gaseous mixture. This apparatus, as shown mounted upon a wooden stand, consists of two glass bulbs blown in a fine glass tube, bent in the manner portrayed in the figure; the lower globe has a larger diameter than the upper, and is capable of holding about 150 c.c. of the reagent employed, while the upper one should be of at least 100 c.c. capacity.

To charge the absorption pipette the liquid reagent is poured into the upper bulb, and the air is then sucked out of the lower bulb through the capillary tube, until the lower bulb is filled with the reagent, which also reaches into the

syphon bend of the capillary tube, but which almost leaves the upper bulb empty.

**Reagents employed.**—A solution of pyrogallic acid and caustic potash in distilled water, *i.e.*, 15 grammes of the acid, and 50 of caustic potash to the litre.

#### THE PROCESS.

1. The amount of atmospheric air is first measured in the gas burette in the following manner :—Both tubes are placed upon a level surface, and distilled water which has been thoroughly shaken up in the air, and thus mechanically saturated with air, is poured down the plain tube A until each tube is about half full. Now, according as the tube A is lowered, will the height of the water in B descend, and *vice versa*. Raise the tube A, therefore, and thus fill the tube B with water ; then lower the tube A, and the atmospheric air of the compartment will rush into the graduated tube B, where it is imprisoned by applying a pinch-cock to the india-rubber tubing at its mouth. This volume of air collected is next exposed to the same atmospheric pressure as obtains in the room, by adjusting the two tubes (on wooden blocks) at the respective heights necessary to insure that the water stands at the same level in each. A reading is then made of the volume of air experimented on, by noting the space (*i.e.*, number of c.c.'s) which it occupies in the graduated tube. It is most convenient to take 100 c.c.

Where the single "absorption pipette," as shown in fig. 36, is employed, the necessary quantity of absorbing reagent is poured in to quite fill the lower and larger bulb ; but for the absorption of oxygen it is necessary to use "a double pipette."

Reagents that are affected by oxygen (such as pyrogallic acid and cuprous chloride) cannot be kept in the single pipette, for the reagent in the lower bulb is exposed to the air. Hempel's double pipette permits of the use of these



reagents without exposing them to the general atmosphere. The double pipette is shown in fig. 37. The first bulb is the

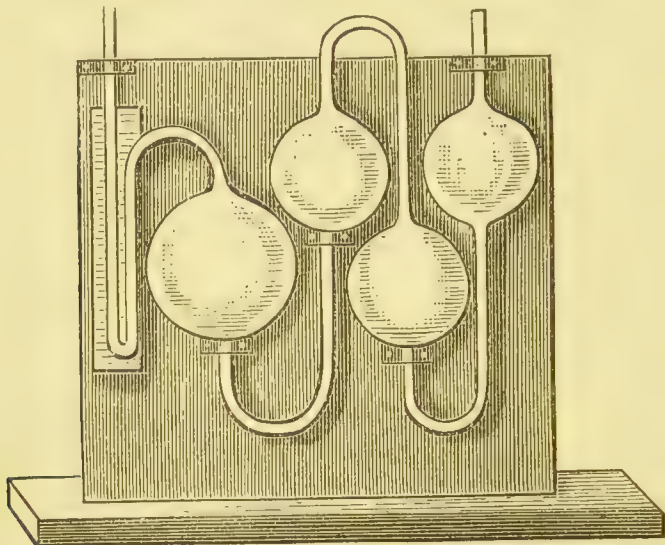


FIG. 37.—Hempel's double absorption pipette.

largest (150 c.c.), and is filled with the absorbent; the next is empty; the third contains water; and the fourth is empty. Thus when the gas is passed into the pipette the water in the third bulb passes into the fourth to make room for the gas, and thus shutting out the atmosphere, the reagent only coming in contact with the small amount of air originally in the second bulb.

A piece of enamel is seen to form a background to a part of the absorption apparatus; this serves to make the liquid in the capillary tube more distinctly visible.

2. Connection is then made, as shown in the figure, by fine stiff india-rubber tubing with the "absorption pipette," which is fitted upon a stand and raised up very close to the tube containing the air, since it is desirable to have as short a length of tubing as possible.

Next, by liberating the two clasps shown in the figure, the gas burette and absorption pipette are put into communication

with each other, and by raising the tube A the water ascends in tube B, and the air is thus forced over into the absorption pipette. The pinch-cock on the burette is closed, the india-rubber tubing is pinched, and a firm clasp applied—after which the pipette may be disconnected and well shaken. After connecting with the absorption apparatus, opening the pinch-cock, and removing the clasp, the residual air can be brought back by lowering the tube A. This is slowly repeated several times, in order to give the solution time to absorb all the oxygen, and the air is finally drawn back into the burette, care being taken that the absorbing solution does not pass beyond the connecting capillary tube. The height of the water in the two tubes is then brought to the same level, just as it was at the commencement of the process (and for the same reason), and then the volume which the air *now* occupies is read off. The volume remaining, when “constant,” is broadly due to the nitrogen, and the difference between it and the original volume represents the oxygen absorbed.

At temperatures about 60 deg. F. the last trace of oxygen is thus removed in about 3 minutes of shaking (Hempel). The solution of pyrogallic and caustic potash will also absorb carbonic acid, sulphuretted hydrogen, sulphurous acid and hydrochloric acid—if present.

Since, as it was pointed out, the conditions of temperature must remain the same throughout the estimation, the gas burette, after it has been charged, should not be handled except by its iron stand, and the apparatus must not be moved about from one spot to another.

The absorbing reagent should always first be saturated by shaking it up with the gases that are but slightly soluble in it, otherwise errors of estimation result, and the necessity of always saturating the water in the burette with the gas under examination must be borne in mind. With care this method of eudiometry gives results but little inferior to those

arrived at by working over mercury and using solid absorbents.

The oxygen may also be estimated by *Dumas' process*, in which the air, having been freed of its carbonic acid by a strong solution of caustic potash, is passed through a combustion tube containing a length of pure spongy metallic copper. The copper is kept ignited, and the air is gradually drawn over the surface of the bright metal, which becomes tarnished by oxidation, and the difference in weight of the original copper and this tarnished metal represents the oxygen taken up from the volume of air experimented upon.

## CHAPTER II.

## COLLECTING SAMPLES. EXAMINATION BY THE SENSES.

SAMPLES are most conveniently collected in large wide-mouthed, glass-stoppered bottles, of about four litres capacity—which, when used for this purpose, are termed “air-jars.” These must be thoroughly cleansed in every case before use—the last washings should be of ammonia-free distilled water. They are then inverted, allowed to drain quite dry, and subsequently stoppered; and after the collection of the sample the jars should be hermetically sealed with lard in those cases where they have to be removed. Lastly, a label which carries a written statement of the current temperature and pressure, is applied.

Following out the principles advocated with regard to water samples, a sample of air should be collected—whether it be vitiated by respiration, combustion, trade-processes, or by products of decomposition, etc.—at the time when, so far as can be judged, the atmosphere will afford its maximum evidence of pollution.

In investigating the case of respiratory contamination in the air of a bedroom, for instance, the sample should be taken shortly before the first riser quits the room; that is to say, after the room has been occupied by its customary number of inmates for the greatest number of consecutive hours.

The proximity of stoves and fireplaces must be avoided, and specimens should always be taken at the mean height at which the air is respired; at the same time a sample

should be taken of the external atmosphere, at the same level, for purposes of comparison.

Samples of furnace gases should be taken from near where the visible flame ends, for beyond this point they may get diluted by air sucked through the porous walls of the flue.

The air is made to occupy the jar by either of the following methods:—

1. A jar may be accurately filled with good water—which can with rare exceptions be got upon the premises, and may with advantage be previously boiled—and then inverted, emptied, and allowed to drain dry in the compartment the air of which is under examination. A sample then rushes in to fill the place of the original water. The water should be about the temperature of the room.

2. The air may be forced in by bellows which are pro-



FIG. 38.—The flexible bellows-pump employed by Angus Smith to draw out air from the air jar.

vided with a nozzle longer than is usual, so that it may be placed well down into the jar to within an inch of the bottom; this insures that the air which originally occupied the jar will be displaced from below upwards in its entirety.

3. The original air in the jar may be pumped out by means



of a small air pump, and if this is done in the atmosphere of which a sample is desired, the jar is, of course, refilled by a sample of such flowing in to fill the void.

Angus Smith drew the air out of the bottle by a flexible bellows pump, shown in fig. 38 (after Angus Smith).

4. A jar may be accurately filled with mercury, and emptied in the compartment where the sample is to be collected. Although this plan is theoretically a good one, it is practically inapplicable on account of the large amount of mercury required, and the difficulty of conveying this (from its enormous weight) from place to place.

The first method is recommended on account of the facility of its execution, and from the fact that it is at least on a par with most of the others as regards its freedom from the possibilities of error.

Whenever it is possible, there is no gainsaying the advantage in greater exactitude attained by making the analysis at once in the compartment in which the sample has been taken, since the general atmosphere is that of the jar; and this can generally be done, although frequently at the cost of much trouble and inconvenience. Sometimes, however, the matter becomes practically impossible, and in those cases the ground glass stopper should be tightly fitted, and then hermetically sealed with lard.

Under all the circumstances and conditions in which we are concerned with the collection and analysis of air, it is apparent how necessary it is for the operator to avoid the error which would be introduced by breathing into the apparatus, or into the reagents, or by handling the jars more than is absolutely necessary with warm hands. There should be as little loss of time as possible in commencing the analysis, and in the meantime the jar should not be exposed to temperatures varying largely from that at which the sample was collected.

In every event the room in which the sample is analysed must be free from draughts and of a uniform temperature, or at least not liable to frequent changes in temperature.

#### EXAMINATION BY THE SENSES.

The extent to which injurious gases are allowed to pollute an atmosphere which is breathed continuously by human beings, is rarely so great as to afford any evidence of their presence save by the sense of smell.

In the case of those whose odour perception is very keen it may safely be said—with regard to odorous gases—that if such are not appreciable by smell they do not exist in injurious amounts. But the sense is remarkably blunt in others, and this fact renders desirable the adoption of further means of detection; but when we consider that many harmful gases are entirely odourless, the adoption of delicate chemical tests becomes imperative.

Fortunately the atmospheric impurities which we are by far the most generally concerned with, viz., organic vapours, etc., from the lungs and skin, give an index of their presence when they have reached a harmful extent, in a manner which is almost universally appreciated. Everyone is familiar with, and can detect at once, the “stuffy” odour in an atmosphere as soon as such organic matter accumulates beyond a certain point—everyone, that is, who passes into such an atmosphere direct from the external air. The atmosphere may, however, be suffered to acquire a high degree of “stuffiness” (with which is synonymous “unhealthiness”), without detection, unless—as is the case with most of our other special senses—we quicken our perception by instituting a comparison (in this case between the external and internal airs).

The perception of odour in the atmosphere is materially influenced by the temperature, the amount of moisture present, and the degree of agitation existing at the time; a low

temperature, little moisture, and perfect quiescence, being all unfavourable to odour perception. The more common gaseous or vaporous impurities which furnish an odour are :— “organic matter,” empyreumatic and tarry matters, ammonium sulphide, sulphuretted hydrogen, ammonia, coal gas, carbon-bisulphide, etc. ; while carbonic acid, carbonic oxide, and marsh gas, are practically inodorous.

*Reaction.*—The air over open country regions and the sea has no evident reaction to litmus papers, and may be said to be neutral. Under those circumstances, however, in which ammonia exceeds its usual amount a faint alkaline reaction may be sometimes produced.

The air of large towns is slightly acid, owing to the sulphurous acid which is derived from the sulphur compounds contained in the substances used for combustion, and the air of occupied rooms in which coal gas is burning is also slightly acid. A piece of delicate blue litmus paper, moistened with neutral distilled water, denotes this acidity by changing in an hour or two to a faint, though distinct, red.

In the neighbourhood of manufactories, it is evident that the air may be markedly acid or alkaline, according to the nature of the predominant gases which escape in connection with the manufacturing process.

## CHAPTER III.

## CARBONIC ACID.

THE estimation of the carbonic acid in the atmosphere is of all air analyses the one of most general importance, and is, on this account, the one most frequently performed. This is not because the carbonic acid is liable to exist in injurious amounts, even under the worst conditions of ventilation commonly obtaining, but because the gas, when furnished by respiration, affords an important clue as to the extent to which another and injurious product is co-existent, *i.e.*, organic matter from the lungs.

So inert is the carbonic acid itself that it may exist, when unaccompanied by this injurious organic agent, to the extent of 1.5 parts per 100, without grave consequences. It is therefore *the carbonic acid which has been added to the atmosphere by respiration*, which mainly, if not solely, concerns sanitarians, and this simply because it affords an index to the amount of organic matter, which, coming from the same source, increases very nearly *pari passu* with it.

The amount of carbonic acid which is present in the general atmosphere—and which may be termed “normal”—has been seen to be 0.04 per cent. by volume; it arises mainly from three sources:—

1. Organic decay, fermentation, and combustion.
2. Animal respiration.
3. The combustion of ordinary fuel, coal-gas, etc.

The lowest estimation of carbonic acid made in any atmosphere was 0.02 per cent.

The purest mountain and sea air contains only 0.03 per cent. Recent investigations tend to prove that carbonic acid exists in greater quantities in the air over the summits of very high mountains than lower down towards their bases.

The external atmosphere, during fogs, often contains 0.07 per cent., and may contain as much as 0.09 per cent.

In an ordinary sitting-room, well lighted by gas, the carbonic acid often reaches 0.2 per cent.

Where there is overcrowding it has been estimated as high as 0.7 per cent., and it is commonly under these circumstances 0.3.

Angus Smith found in the worst parts of theatres 0.32 per cent.; in mines, he found an average of 0.785, but in one case the amount reached as high as 2.5 per cent.

Fatal results would not accrue with less than 5 to 10 per cent.

We are indebted to Pettenkofer, of Munich, for a method of estimating the amount of this gas which, owing to the facility of its performance, is very generally adopted.

So small is the amount of carbonic acid existing in the atmosphere under even bad conditions of ventilation (save in mines), that eudiometry fails to indicate its amount, and resort is had, in Pettenkofer's process, to make the gas combine with the baryta of baryta water ( $\text{BaO} + \text{CO}_2 = \text{BaCO}_3$ ); and then to estimate the quantity thus absorbed by the diminished alkalinity which the acid has effected in the baryta water.

#### PETTENKOFER'S ALKALIMETRIC METHOD OF ESTIMATING THE CARBONIC ACID IN THE ATMOSPHERE.

The rationale of the process is as follows:—Clear baryta water—an alkaline medium—will absorb carbonic acid with great readiness, and thereby become turbid ( $\text{BaO} + \text{CO}_2 = \text{BaCO}_3$ ); the carbonic acid will diminish on this account



the degree of alkalinity (the "causticity") of the original baryta water, according to the extent to which it is absorbed. If, therefore, the degree of alkalinity of a measured quantity of baryta water is estimated, and then this reagent be made to take up all the carbonic acid of a sample of air, the diminished alkalinity of the baryta water—when subsequently tested—will represent the amount of carbonic acid which has combined with the BaO (baryta).

**Apparatus required for Pettenkofer's method :—**

1. A large air-jar, *i.e.*, a large wide-mouthed glass stoppered bottle of about 4 litres (4000 c.c.) capacity. It is necessary to find the exact capacity of the bottle in order that the amount of air which it will hold may be accurately known. This can be ascertained by filling the bottle quite full of water, and then measuring the water as it is emptied out—the volume of the water which the bottle holds will likewise be the volume of air which takes its place.

2. A white porcelain dish.

3. A glass stirring-rod.

**Chemical reagents :—**

1. Pure clear baryta water, 4.5 grammes of the crystallised hydrate to the litre.

2. A standard solution of oxalic acid (crystallised) made to such a strength (*i.e.*, 2.863 grammes to the litre) that 1 c.c. corresponds in acidity to 0.5 c.c. of carbonic acid.

3. A solution of phenol-phthalein (1 part in 250 parts of alcohol).

**THE PROCESS.**

1. A sample of the air should be collected in the air-jar.  
2. 50 c.c. of perfectly clear baryta water are then placed in this, and the liquid is occasionally made to flow round the sides of the jar by rolling it about on its side for a minute or two. The air is kept in the jar for at least an hour—in order

that time may be given for *all* the carbonic acid present in the sample to combine with the baryta, and thus create a turbidity of the carbonate of baryta.

3. The alkalinity of 25 c.c. of the clear baryta water is meanwhile tested in the following manner:—The 25 c.c. are placed in a small flask, and are tinted pink by a drop of phenol-phthalein, and the standard solution of oxalic acid is cautiously run in from a graduated burette, until the alkalinity of the baryta water has been just neutralised by the acid; then the amount of acid necessary to effect this neutrality is read off from the burette. The exact stage when neutrality is reached, is indicated by the disappearance of the pink colour created by the phenol-phthalein. Care must be taken not to add a drop of acid beyond this point, or the causticity of the baryta water will be over-estimated.

4. At the end of an hour, 25 c.c. of the baryta water are removed from the air-jar, and the causticity is estimated in a precisely similar manner, but in effecting the removal the precipitate of barium carbonate which has settled must not be disturbed.

5. The difference in the number of cubic centimetres of acid solution required to neutralise (*a*) the original baryta water, and (*b*) that which has taken up the carbonic acid of the air in the jar, represents half the amount of carbonic acid in the air—since another 25 c.c. of baryta water remains in the jar, and this has been weakened by carbonic acid to a similar extent to the 25 c.c. which has been removed and tested.

6. The sample of air examined must not be counted as 4000 c.c., for when 50 c.c. of baryta water were added to the jar an equivalent bulk of air was displaced. The air experimented upon therefore represents  $4000 - 50 \text{ c.c.} = 3950 \text{ c.c.}$

7. The result may be returned as the amount of carbonic acid per cent. (or per 1000 parts) of air, at the “current temperature and pressure,” after carefully noting what these

respectively were at the time of experiment. Or, as is more usual, it may be reduced to the "standard temperature," *i.e.*, 32 deg. F., and the "standard pressure," *i.e.*, 29.992 inches, or 760 millimetres, of mercury.

To reduce results to the standard temperature of 32 deg. F., it is necessary to bear in mind the fact that air expands, when heated, to the extent of 0.002 of its bulk for every degree rise above 32 deg. F., and in consequence at this temperature a larger amount of cold condensed air would occupy the same air-jar than would be the case with the warmer and more expanded air at 68 deg. F. Supposing that we have found that the carbonic acid represents 0.07 per cent. per volume of the sample of air collected when the current temperature was 68 deg. F., and we require to find what percentage amount by volume this will correspond to at 32 deg. F. Since our estimation is made of 100 vols. at 68 deg., it is evident that at the lower temperature of 32 deg. the air will contract to a bulk which is less than 100 vols. at 68 deg. How many vols. of air, then, should be taken at the current temperature (68 deg.) in order to represent 100 vols. at 32 deg.? There is in this case a difference of 36 deg. between 68 and 32, and for each of these degrees the air will contract 0.002 of its volume; that is to say, each volume of air at 68 deg. will become  $1 - (36 \times 0.002) = 0.928$  at 32 deg. and 100 volumes will become 92.8. Now if 100 vols. of air at 68 deg. represent 92.8 at 32 deg., how many vols. at 68 deg. will represent 100 volumes at 32 deg.?  $92.8 : 100 :: 100 : x = 107.75$ .

It is necessary, then, to find the amount of carbonic acid in 107.75 volumes at the current temperature, in order to ascertain the amount which will be present in 100 volumes at 32 deg. F.

But 100 volumes at 68 deg. contain 0.07 of carbonic acid, and therefore 107.75 volumes contain:—

$$100 : 107.75 :: 0.07 : x = 0.075.$$

That is to say, since 100 volumes of the air at 32 deg. correspond to 107.75 at 68 deg., 100 volumes at 32 deg. will contain 0.075 parts of carbonic acid.

The correction to the standard barometric pressure is not necessary unless the experiment is made upon a considerably elevated region; for the circumstances under which a sample is collected and analysed for public health purposes, the correction need rarely, if ever, be made, and at best must represent an insignificant figure. The object is to reduce the percentage amounts of carbonic acid at the current pressure to the percentage amounts at the standard pressure, since air occupies a smaller or larger volume, according to the pressure which it is subjected to. The correction is made upon the same lines as that of temperature, when necessary, by the following estimation:—

As the current height of the barometer : the standard height (29.922 inches) :: the percentage amount of carbonic acid at the standard temperature, and at the current height : that of the standard height and temperature.

Suppose, for instance, the carbonic acid at the barometric reading of 29.5 inches is 0.075, then:—

$$29.5 : 29.922 :: 0.075 : x = 0.076.$$

The amount is seen to be greater, since more air would be compressed into the 100 volumes by the greater atmospheric pressure.

*Example.*—The causticity of 25 c.c. of the original and clear baryta was tested by cautiously running in the standard acid solution.

20 c.c. of the acid solution were required to just effect neutralisation.

25 c.c. of the baryta water from the air-jar required only 17 c.c. of the acid solution to neutralise its diminished causticity.

Therefore  $20 - 17 = 3$  c.c. of the acid solution repre-

sents the carbonic acid taken up by this baryta water from the sample of air.

But 1 c.c. = 0.5 c.c. of carbonic acid.

$\therefore$  3 c.c. = 1.5 c.c.                    "                    "

The other 25 c.c. of baryta water left in the jar will also have combined with another 1.5 c.c. of carbonic acid.

$\therefore$  The carbonic acid combined—or, what is the same thing, the carbonic acid originally in the air-sample, = 3 c.c. The capacity of the jar was 4000 c.c., and the air examined is 4000 c.c. — 50 c.c. (space occupied by the lime-water) = 3950 c.c.

$\therefore$  There are 3 c.c. of carbonic acid in 3950 c.c. of air, or  $3950 : 100 :: 3 : x$  carbonic acid per cent. = 0.0759—at the current temperature and barometric pressure (*i.e.*, 72 deg. F. and 29.8 inches).

#### NOTES UPON THE PROCESS.

The estimation is a very approximately correct one, and quite near enough for all practical hygienic purposes.

1 c.c. of  $\text{CO}_2$  at 32 deg. F. weighs 1.96633 milligrammes at 760 m.m. pressure; the relation, therefore, between the volume and the weight =  $\frac{1}{1.96633} = 0.508$ .

In order that the baryta water shall be kept quite pure it will be necessary to remove the carbonic acid from the air which enters the store bottle when some of its contents are withdrawn—as by making it pass through the pumice stone moistened with caustic potash. The accompanying figure will serve to demonstrate how this can be readily effected:—A large glass store bottle is represented fitted with a glass tap to draw off the clear baryta water; any air which enters must pass through the U-shaped tube, which is packed with the pumice moistened with caustic potash solution.

A much more recent method than Pettenkofer's, and one



which has the recommendations of easier and quicker accomplishment, is **the process of Lunge and Zeckendorf**. In the writer's opinion the process is not so precise and reliable as Pettenkofer's, and it is least satisfactory where the

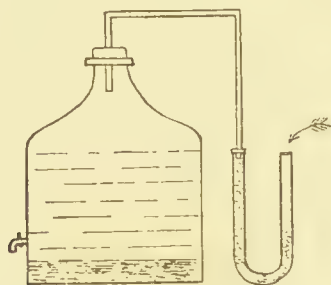


FIG. 39.—Store bottle for baryta water.

amount of  $\text{CO}_2$  is very low, but it is a useful means of readily making a closely proximate estimation.

In this method a decinormal solution of soda is prepared (5.3 grammes of anhydrous carbonate to the litre), to which a little phenol-phthalein is added—a purple solution resulting which keeps well.

A small caoutchouc bag of 70 c.c. capacity, and fitted with two valves working opposite to each other, so that on pressing it the air is forced in one direction only, is connected by means of an india-rubber tube with a glass tube, and this passes through a perforation in an india-rubber cork fitted tightly to a small flask of known capacity. The bag is first pressed several times so as to fill it and the bottle with the air to be tested, and then 2 c.c. of the decinormal soda are added to 100 c.c. of freshly distilled ammonia-free water, and 10 c.c. of this  $\frac{1}{500}$  normal solution are emptied into the small flask.

The air of the bag is slowly pressed over into the reagent, the flexible tube is tightly compressed with the fingers, and the flask is well shaken. This is repeated with another bag-full of air, and so on until the purple colour is discharged.

Lunge found, by direct experiment, the percentage amount of carbonic acid which corresponds to the number of times

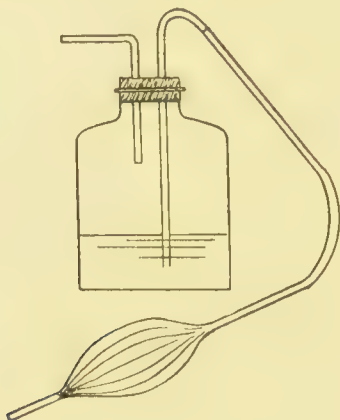


FIG. 40.—The apparatus for the Lunge and Zeckendorf process.

the contents of the ball are emptied into the bottle. His results were as follows:—

Number of Pressures of India- rubber Ball.	Parts per cent. of Carbonic Acid in the Air.	Number of Pressures of India- rubber Ball.	Parts per cent. of Carbonic Acid in the Air.
2	0'30	15	0'074
3	0'25	16	0'071
4	0'21	17	0'069
5	0'18	18	0'066
6	0'155	19	0'064
7	0'135	20	0'062
8	0'115	22	0'058
9	0'10	24	0'054
10	0'09	26	0'051
11	0'087	28	0'049
12	0'083	30	0'048
13	0'08	35	0'042
14	0'077	40	0'038
		48	0'030

The inverse method of collecting a measured volume of air, and adding to it the standard soda and indicator until the colour changes, well shaking after each addition, is easy of application and gives good results.

**Schaffer** and **Wolpert** moisten a piece of blotting-paper with standard solution of sodium carbonate and indicator (phenol-phthalein), and suspend it in the air; the time which elapses before the paper loses its pink colour gives the indication of the amount of  $\text{CO}_2$  in the atmosphere. The varying degrees of air-movement in the room lead to error.

The writer prefers to make the estimation of carbonic acid in a long cylindrical jar of the capacity of about 2 litres, fitted on the top, but to one side, with a doubly perforated cork—one perforation transmitting the drawn-out point of a graduated burette with stop-cock, and the other perforation a small piece of glass tubing carrying externally a small close-fitting india-rubber cap. The air is collected, as directed; as much as 200 c.c. of baryta water (standardised at the time of use by decinormal oxalic acid) is added through the glass tube, which is then sealed by its cap. After one hour, the bottle being gently rolled along a table from time to time in the meanwhile, a few drops of phenol-phthalein are added through the small glass tube—the cap of which is removed for good so soon as the burette has been charged with the decinormal oxalic acid solution and this is allowed to discharge cautiously into the bottle. So soon as the pink colour commences to weaken, the acid is added, drop by drop, and the bottle and its contents gently shaken and stood upon a white porcelain dish. The shape of the bottle and the extra depth of baryta water favour the delicacy with which the end change can be noted, when the experimenter looks down from above on to the white porcelain dish.

*Conclusions to be drawn from the amount estimated.*—It is generally agreed that the air of compartments that are

occupied by human beings should not show more than 0.06 per cent. of carbonic acid, *i.e.*, the 0.04 per cent. which exists normally in the air, and 0.02 per cent. of "allowable respiratory impurity." This limit is chosen because, when it is reached solely from respiration, the organic matter, etc., is judged to have reached an unhealthy amount, and is said to be appreciable to the sense of smell (creating a "stuffy" atmosphere). From the writer's experiments nothing is generally thus appreciated until the carbonic acid reaches 0.08 per cent., in those cases where samples have been collected from rooms occupied under ordinary conditions.

It is a question, however, to what extent this stuffiness is due to exhalations from the skin, and as to whether the headache which it induces is not due to causes other than the organic matter from the lungs, which is, according to many experiments, certainly not so poisonous as it is generally said to be.

When the carbonic acid from respiration carries the total up to 0.15 per cent., many of the inmates will then complain of headache, torpitude, lassitude, and faintness; and no further impurity can be detected by the sense of smell after the carbonic acid, from respiration, has carried the total up to 1.3 per cent.

It is, of course, not necessary that rooms in which gas lights are burning shall show only a percentage amount of carbonic acid equivalent to 0.06 per cent., since in these cases there is no concomitant organic matter, such as is given out by the lungs. The extent to which a common gas burner may furnish carbonic acid to the atmosphere is about ten times that of an average adult, *i.e.*, about six cubic feet per hour; and the amount of sulphur compounds thus yielded in a gas well purified is not important hygienically.

The air over burial grounds, especially when these are crowded, is said to contain an abnormally high amount of carbonic acid.

**Angus Smith** devised a rough “household test,” which, though little adopted at the present time, is simple and practical, and as such is of special value for determining as to whether the carbonic acid is above or below “the allowable limit.” He found, by experiment, the largest volume of air which could be shaken up with clear lime-water without furnishing a turbidity so long as the carbonic acid therein did not exceed the allowable amount, viz., 0.06 per cent., and from the findings of his experiments he propounded the following: “Let us keep our rooms so that the air gives no precipitate, when a 10½ ounce bottle is shaken up with half an ounce of clear lime-water”; which is equivalent to stating “let us keep our rooms so that the carbonic acid shall not exceed 0.06 per cent.”

His results are set forth in the following table:—

Size of bottle in ounces.	Size of bottle in cubic centimetres.	Volume of air in cubic centimetres.	Carbon dioxide in the air per cent.	Size of bottle in ounces.	Size of bottle in cubic centimetres.	Volume of air in cubic centimetres.	Carbon dioxide in the air per cent.
20.63	584	571	.03	6.00	170	156	.11
15.60	443	428	.04	5.53	157	143	.12
12.58	356	342	.05	5.15	146	132	.13
10.57	299	285	.06	4.82	137	123	.14
9.13	259	245	.07	4.53	128	114	.15
8.05	228	214	.08	3.52	100	86	.20
7.21	204	190	.09	2.92	83	69	.25
6.54	185	171	.10	2.51	71	57	.30



## CHAPTER IV.

## THE ORGANIC MATTER IN THE AIR.

THE organic matter in the air includes that given off from the lungs and skin, and this varies in character and amount in different human beings, with the constitution and state of health of the individual. It probably forms pabulum for the nourishment of germs, while at the same time its presence debilitates those exposed to it and renders them more susceptible to diseases and less capable of combating them. Its composition is very imperfectly understood; it probably consists partly of volatile fatty acids and their ethers, and partly of vaporous and suspended matters (epithelial and fatty debris). It is certainly largely nitrogenous and oxidisable, since it will deoxidise solutions of the permanganate of potassium and will yield ammonia. It quickly putrifies; and when air containing it is aspirated through sulphuric acid the organic particles are charred and darken the solution. When present in large amounts in water it can be precipitated by silver nitrate. Probably the major part is molecular and suspended, since it does not diffuse equally about a room, and tends to fall and settle; and there is no doubt that it is mostly in combination with watery vapour, for substances absorb it according to their hygroscopic powers (*i.e.*, it is absorbed chiefly by wool, feathers, etc., and least so by horse-hair). It gives a foetid smell to the atmosphere, and from the persistence of this smell it is doubtless burnt off but slowly by the atmospheric oxygen; and in small quantities it gives odour to water.

*The processes employed for the estimation of this matter*

in air are preferably those with which we have already grown familiar, and which have served to detect the same matter in water. A large measured volume of the air is made to slowly pass through doubly distilled ammonia-free water, which will retain all the soluble and suspended material—including organic matter as such, and those gases which afford evidence of the presence, or former presence, of such matter. The water is then tested by Wanklyn's method—as to its nitrogenous organic matter, and by Tidy's improvement of the Forchammer process—as to its oxidisable organic matter; it being borne in mind in the latter test that either nitrous acid, sulphurous acid, sulphuretted hydrogen, or tarry matters, will, if present, also decolorise the permanganate.

The most convenient *method of performance* is to take a glass wash-bottle, partially fill with 500 c.c. of doubly distilled ammonia-free water, and then tightly fit with a doubly perforated india-rubber cork. Into one perforation a glass tube bent at right angles, with one trumpet-shaped extremity, is accurately fitted, with the trumpet-shaped end projecting externally (to collect the air), while the other extremity is made to dip well down into the distilled water; the second perforation conducts another bent glass tube, the end contained within the flask being above the surface of the water, and the other connected directly by india-rubber tubing to the aspirator. The capacity of the aspirator being known (and a convenient size is that of 25 litres), it is filled to the top with tap-water, the tap is then turned so that the water passes slowly out—when air enters the trumpet-shaped mouth of the bent glass tube to take the place of the escaping water; such air is washed in the distilled water (which has the property of taking up gases, etc., with extreme readiness) before it reaches the aspirator.

*Example.*—If it is desired to make an estimation of the

nitrogenous organic matter the aspirator is twice filled and allowed to empty, 50 litres of air will have replaced the 50 litres of water, and these will have been drawn through the

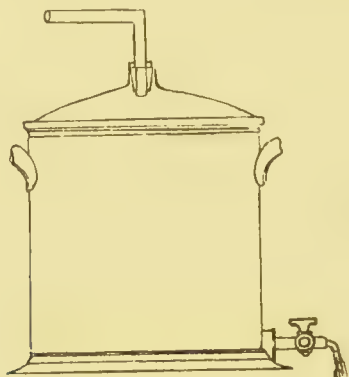


FIG. 41.—An aspirator.

500 c.c. of distilled water; therefore this 500 c.c. of water will contain the nitrogenous organic matter of 50 litres of air.

Suppose the 500 c.c. of water are found to contain by Wanklyn's method 0.05 milligramme of albuminoid ammonia, then there will be 0.05 milligramme of such ammonia in 50 litres of air. But in dealing with air the results are expressed in terms of "milligrammes per cubic metre" (*i.e.*, 1000 litres).

Therefore, if there is 0.05 milligramme of albuminoid ammonia in 50 litres, there will be 1 milligramme of free ammonia in 1000 litres—or a cubic metre—of air.

Generally speaking the external atmosphere averages about 0.06 milligramme per cubic metre of *free ammonia*, but this has been estimated as high as 0.8 in hospital wards.

Such air contains *albuminoid ammonia* up to 0.1 milligramme per cubic metre, and averages about 0.08. In a hospital ward this ammonia has been estimated at 1.3.

The process is sometimes performed by passing the air through a succession of small wash bottles containing am-

monia-free distilled water—instead of one large bottle. The results will be found to be the same in either case.

The *oxidisable* organic matter may also be thus collected ; but before estimating this, any sulphuretted hydrogen, sulphurous and nitrous acids, and chlorine compounds, must be disposed of or accounted for in the manner already shown in water analysis ; any tarry matter will generally give evidence of its presence by yielding a smell or turbidity to the water.

The water may also be tested for oxidised nitrogen (nitrates and nitrites), chlorine, etc., as in water analysis.

Another, rougher, plan is to surround a thoroughly clean U-shaped glass tube with a freezing mixture of salt and snow (or ice), and to aspirate the air through the tube, when the low temperature condenses the watery vapour in the air as it passes through, and most of the organic matter is thereby collected. The interior of the U-shaped tube can then be washed with ammonia-free distilled water, and the washings treated for “free” and “albuminoid” ammonia, etc.

This plan may also be used for the collection and examination of suspended matters.

Another method of estimating the organic matter in air is that of Carnelly and Mackie. In this process from 3 to 4 litres of air are shaken with 50 c.c. of normal solution of potassium permanganate for five minutes, and the amount of decomposed permanganate is deduced, on colorimetric principles, from the loss in colour sustained by the original solution ; and from the extent of this loss, as estimated by the amount of standard solution required to restore it, the amount of oxygen absorbed can be calculated (1 c.c. of the normal solution = 0.008 gramme  $O_2$ ).

## CHAPTER V.

AMMONIA—MARSH GAS—CARBONIC OXIDE—SULPHUR COM-  
POUNDS.

## AMMONIA.

TRACES of ammonia are present in every atmosphere. In towns it generally amounts to about 0.06 milligramme per cubic metre. Such traces are derived from organic decomposition, from combustion (coal), and from respiration. The ammonia exists generally in combination with an acid, as the carbonate and chloride, and, less commonly, as the nitrate or sulphate. Ammonia vapours do not appear to injure health beyond affecting the conjunctiva when in excessive quantities, and it is doubtless one of the most wholesome forms in which nitrogen and hydrogen, as gases, pass into the air. It is in greatest quantity near the ground, over peaty land it is abundant during hot weather, and it is largely present at the back of houses where refuse matter is deposited. Its presence, however, is regarded with scant favour, in spite of its comparative innocuousness, for it is a product of decomposition, and it is a constant ingredient in the most impure airs.

A large amount of ammonia in the atmosphere may be detected by moistening strips of filtering paper with Nessler's reagent, and hanging these up for some time in the air of the compartment; or if a sample of air is collected in a 4-litre jar—by catching one of these prepared papers between the stopper and the neck in such a way that it hangs down into the jar free of the sides for a few hours. But when present only as



faint traces, large quantities of air must be aspirated through doubly distilled ammonia-free water, and the ammonia tested for by Nessler's reagent ; and if the air be measured it may be estimated quantitatively by "Nesslerisation."

### MARSH GAS ( $\text{CH}_4$ ).

This gas probably exists in traces in most atmospheres, although owing to the difficulties of its detection in faint traces it is not easy to speak definitely upon this point. There are certainly traces in the atmosphere of towns, and over districts of abundant vegetation (especially when such districts are marshy) it exists in large quantities. As it is evolved from strata in which mining operations are progressing it is known as "fire-damp" ; and its character of exploding when ignited in the presence of carbonic acid is often disastrously exemplified.

There is no doubt that marsh gas, though apparently extremely innocuous, may create after a while symptoms of poisoning ; and being inodorous and non-irritating its presence would not be detected by the senses. Any escape of coal-gas, containing as it does 32 per cent. of marsh gas, will charge the atmosphere with considerable and dangerous amounts of this substance, but fortunately in these cases the strongly smelling ingredients of the coal gas give timely warning.

### CARBONIC OXIDE ( $\text{CO}$ ).

Owing to the properties which this gas possesses of entering into combination with the hæmoglobin of the red corpuscles, displacing their oxygen, and thus paralysing their oxygen-carrying functions, it plays the part of a virulent narcotic poison ; and since it gives no indication of its presence to the sense of smell its opportunities for evil are materially

enhanced. It becomes, then, an important matter to examine the air for this gas in those cases where there is any likelihood of its presence, and this will always be the case in the atmosphere of compartments where iron or copper stoves are employed, and especially so when the material is cast iron and when the fuel is coke; where coal gas (which contains 7 per cent.) is incompletely burnt or escapes; or where there is a possibility of some of the products of combustion from furnaces, flues, etc., escaping into a compartment—for the air in furnace-flues has been found to contain over 20 per cent. of carbonic oxide, and that of ordinary flues from domestic fire-places as much as 4 per cent. The carbonic oxide of the air of flues is always the product of imperfect combustion, that is to say, the carbon of the organic material burnt is either not fully oxidised to carbonic acid ( $\text{CO}_2$ ) owing to the supply of fresh air being insufficient, or else the carbonic acid, being formed low down in the furnace, gets reduced in passing through the rest of the furnace to carbonic oxide.

Of the gases generated from the explosion of gunpowder, carbonic oxide forms nearly 8 per cent. (7.5 per cent.); and a serious drawback to the adoption of "water gas" as a source of heat and light, is the fact that it contains (before combustion) 25 to 40 per cent. of this very dangerous ingredient.

The carbonic oxide in the atmosphere of stove-heated rooms is derived from either of the following sources (and probably to some extent from all):—

1. Red-hot cast iron will transmit the gas from the fire—either through its substance, or through minute fissures; and the hot iron will even itself reduce  $\text{CO}_2$  to  $\text{CO}$ .

2. The carbon which enters into the formation of the cast iron may get oxidised, and reach the external atmosphere.

3. Particles of suspended organic matter in the atmosphere get charred and partially oxidised by coming in contact with the heated stove.

4. Currents passing down the smoke flue, as they do under certain conditions, may thus introduce the gas.

#### QUALITATIVE TEST OF ITS PRESENCE.

The best is Vogel's—but a knowledge of the spectroscope is necessary to the test, and for those unacquainted with the use of this instrument a short introductory description is given:—

If a compound light, such as sunlight, is made to pass through a glass prism, the different coloured rays of which it consists are unequally refracted (or bent out of their original course), so that beyond the prism they form, upon a white surface, a continuous line of colours called the “spectrum”; and the spectrum of the compound white light will be seen to consist, in order, of red, orange, yellow, green, blue, indigo, and violet. A number of dark lines—called absorption bands, or Fraunhofer's lines—are also seen to cross the image of the solar spectrum. Each of these lines occupies a definite position, and therefore affords a means of accurately localising other lines.

In other lights, however, the spectrum will only show a very few *bright* bands (that of the sodium flame only *one*), and the remainder of the spectral image is thus rendered almost—or quite—invisible by comparison.

If now we transmit solar light through different coloured solutions, we then get different absorption bands. If a solution of fresh blood, for instance, be taken, and a small colourless cell containing it is placed before the slit in the instrument which admits the light, two distinct and characteristic dark stripes or absorption bands appear.

The accompanying figure will serve to show the manner in which the spectroscope is constructed.

A firm iron stand is seen to support at its upper end a brass plate carrying the glass prism; laterally a cylinder is also

fastened to the brass plate, and in the end of this cylinder which is nearest the prism is placed a lens, the other end being closed by a plate with a vertical slit in it (the breadth of which can be regulated by a screw to meet requirements); through

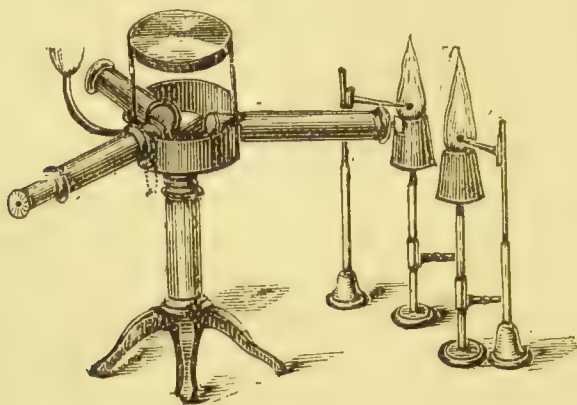


FIG. 42.—The spectroscope.

this slit the light (represented by two coloured flames in the figure) is admitted to the prism—the rays being first rendered parallel, and condensed, by passing through the lens. The spectroscopic appearance is then viewed through a small telescope (with a magnifying power of 8), and this is fitted on to the cast iron foot so as to be moveable in a horizontal plane about the axis of the foot. The other moveable tube seen in the figure carries a photographic copy of a fixed scale, which is reflected from the front surface of the prism into the telescope, and serves the purpose of fixing the width and distance apart of the various characteristic lines—it is illuminated by a small gas flame, as shown in the figure. This illuminated scale is now generally dispensed with, the telescope being made to move over a scale which can be read with a Vernier.

All foreign light must of course be cut off, and this is done by a black cloth which is thrown over the prism and the tubes.

The slit is also furnished with a reflecting prism, by means of which two spectra can be compared at the same time.

Thus by a spectroscopic examination, the colour, number and position of the bright lines on the spectral scale are carefully observed and noted. If it is desired to distinguish metals by means of their spectral lines, the substance is dissolved in a drop of the purest hydrochloric acid. A piece of recently ignited platinum wire is dipped in the solution and then held in the Bunsen flame. Coloured liquids are placed in small colourless glass cells fixed on a support and placed immediately in front of the slit, moving them a little until the absorption bands become most distinct.

The most convenient and delicate method of performing spectroscopic observations is by means of the Sorby-Browning micro-spectroscope, which simply consists of a small spectroscope placed in connection with a microscope in such a way that the former fits into the tube of the latter in the same manner as an eye-piece.

*Vogel's test* is so delicate that it more than suffices for all practical purposes, detecting as it does as little as 0.03 per cent. Into a wash-bottle a little pure water is emptied; and the finger is then pricked so that a drop or two of blood may be made to fall into the bottle, which is afterwards connected to an aspirator. At least 10 litres of air are then drawn through the faintly reddish liquid. The whole is then rolled about for half an hour and allowed to stand for a short while, when a little of the reddish liquid is removed and examined by the spectroscope, and the appearance on the scale is carefully noted.

Oxidised hæmoglobin shows two well marked bands in the yellow and in the green parts of the solar spectrum, both lying between Fraunhofer's lines D and E. The spectral appearance of hæmoglobin in the presence of carbonic oxide is almost identical, but the left hand band (at yellow end) of



the carbonic oxide hæmoglobin lies a little nearer to the right (blue end) than in the case of oxy-hæmoglobin. The blood also takes up a more or less marked bluish tint.

Two drops of a colourless solution of ammonium sulphide are next added, the bottle is well shaken, and the liquid is re-examined. If no marked change in the spectroscopic appearance of the fluid has ensued, carbonic oxide *is present*; otherwise the ammonium sulphide having de-oxidised or reduced the hæmoglobin, the two bands will be represented

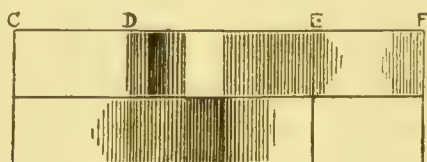


FIG. 43.—Showing the characteristic disposition of the absorption bands, in the spectroscopic picture of oxy- and reduced hæmoglobin. The upper scale representing oxy-hæmoglobin and the lower reduced hæmoglobin.

by a single band shaded off at the borders, and occupying a position about intermediate with regard to the original two bands.

The most delicate results are obtained by placing a mouse in a wire cage and allowing it to breathe the air of the room for several hours. The mouse may be drowned in its cage and the blood then examined by the spectroscope. A control test may be made from the blood of a mouse which has not been exposed to carbon monoxide.

Welzel has devised a delicate chemical test for carbon-monoxide hæmoglobin:—To 10 c.c. of the solution of blood he adds 5 c.c. of a 20 per cent. solution of potassium ferrocyanide, and 1 c.c. acetic acid (1 vol. of glacial acetic acid to two vols. of water); the precipitate very soon becomes reddish-brown if carbon-monoxide hæmoglobin be present, but

greyish-brown with oxy-hæmoglobin—the difference slowly disappearing.

#### A. QUANTITATIVE ESTIMATION.

The subchloride of copper (made by exposing copper turnings and the oxide of copper to the action of strong hydrochloric acid—S.G. 1.124) has the property of absorbing this gas; and advantage may sometimes be taken of this fact to estimate the quantity present by the method of eudiometry.

It will be unnecessary to repeat here what has already been described in connection with oxygen and nitrogen; it will suffice to state that a volume of air is measured in the Hempel's burette (at the current temperature and pressure), the  $O_2$  and  $CO_2$  being first removed, and then the residual air is passed over, slowly and repeatedly, into a double absorption pipette charged with the solution of subchloride of copper.

It is necessary to use an absorption apparatus with large bulbs, in order that a good quantity of the copper solution may be employed; time must be allowed for complete absorption, which takes place slowly; two or three treatments should be repeated until a "constant reading" is obtained, to ensure that all the gas has been absorbed. If there is a marked amount of carbonic oxide present, the loss in the original volume, taken under the same conditions of temperature and pressure, is appreciable, and is due to carbonic oxide, and represents its amount; or the cuprous chloride solution may be transferred under suitable precautions, and boiled in vacuo, and the expelled gas collected and analysed; quite 98 per cent. of the carbonic oxide actually present will be obtained by the latter method.

The faintest traces will escape detection (as in the case of carbonic acid) by this method of eudiometry. The union of cuprous chloride with carbonic oxide is very feeble, and the solution readily parts with the carbonic oxide to the atmos-

phere on shaking; the solution will also absorb acetylene and ethylene.

An amount of under one per cent. has caused narcotic poisoning, and traces far short of this must of necessity, from the nature of the gas, work insidious harm to animal life. It seems, therefore, that the qualitative test of Vogel's, which will readily detect 0.03 per cent., will well suffice for hygienic purposes—since it is not necessary to estimate the *amount*, when even such a faint trace suffices to make an atmosphere very undesirable.

**Sulphurous and sulphuric anhydride, sulphuretted hydrogen, and ammonium sulphide** may all be present in the atmosphere of large towns, the first two invariably so, in traces; the latter two are, however, less often appreciable. They are also added to certain atmospheres by combustion, decomposition and trade processes.

There is no doubt but that their presence is deleterious to health, and creates in time a low unhealthy condition; their presence is, moreover, unfavourable to vegetation.

The external atmosphere chiefly gains sulphur compounds from the combustion of the coal used in manufactories and dwellings—especially where this is impure and contains quantities of pyrites.

Angus Smith considers sulphuretted hydrogen “one of the most deadly of gases,” and holds that “it lowers the tone of health, and may gradually diminish vitality to such an extent that disease ensues”; certainly this may be so when it gains access to the atmosphere, as it sometimes does, through organic decomposition.

Sulphurous acid in large quantities appears to favour the development of—even if it does not induce—chronic chest conditions, anæmia, and conjunctivitis, etc. A cubic metre of air examined at Lille was found to contain on an average 1.8 cubic centimetres. It may be estimated by aspirating

a known quantity of the air through a dilute solution of bromine in water, and precipitating the sulphuric acid thus formed by barium chloride solution; or by absorbing in 20 per cent. potassa lye and titrating with decinormal sodium thiosulphate (24.8 grammes to the litre), after absorption in decinormal solution of iodine (12.65 grammes to the litre) ( $\text{SO}_2 + \text{I}_2 + 2\text{H}_2\text{O} = \text{H}_2\text{SO}_4 + 2\text{HI}$ ); 3.2 milligrammes  $\text{SO}_2$  decolorise 1 c.c. of the iodine solution.

Sulphuretted hydrogen and ammonium sulphide may be detected by exposing to the air strips of filtering paper moistened with a solution of lead acetate. Any faint evidence of darkening about the borders of the previously white paper will prove the presence of these gases in the atmosphere. If the darkening is due to ammonium sulphide the air will at the same time be alkaline in reaction, and similar paper moistened with the blue solution of the nitro-prusside of sodium will turn violet.

A quantitative examination of sulphuretted hydrogen may be made by drawing a known volume of air through a dilute freshly-prepared decinormal solution of iodine in iodide of potassium, to which some starch paste has been added. The operation is stopped as soon as the solution becomes colourless ( $\text{H}_2\text{S} + \text{I}_2 = 2\text{HI} + \text{S}$ ). Each c.c. of the iodine solution decomposes 1.7 milligramme  $\text{H}_2\text{S}$ .

In the case of ammonium sulphide the violet colour produced by the nitro-prusside of sodium may be titrated.

**Hydrochloric, nitric and nitrous acids** favour chronic chest conditions; and **carbon bisulphide** induces serious chronic nervous derangements. Large volumes of the air should be passed through distilled water—which may be afterwards tested for hydrochloric, nitric, and nitrous acids, as in Water Analysis (*quod vide*). Hydrochloric acid may advantageously be absorbed in 10 per cent. soda lye (free from sodium chloride), and then titrated after neutralisation of the

solution by dilute nitric acid. The amount of nitric acid in air is very small ; it is most marked after thunderstorms and in the air of towns. When present in marked amount it may be estimated eudiometrically by absorption in a solution of ferrous sulphate, and nitrous acid may be estimated by combustion with hydrogen and oxyhydrogen gas, the nitrous acid being thereby split up into water and nitrogen (Bunsen). Chlorine and bromine may be absorbed in a pure 10 per cent. colourless solution of potassium iodide ( $\text{Cl}_2 + 2\text{KI} = 2\text{KCl} + \text{I}_2$ ) and the liberated iodine titrated by decinormal sodium hyposulphite, with starch :  $12.65$  milligrammes  $\text{I}_2 = 8.00$   $\text{Br}_2 = 3.53$   $\text{Cl}_2$ .

*Table of the extent of gaseous impurity which has been shown to injure health* (compiled from the investigations of Lehmann, Matt, Gruber, Ogata, and Friedländer).

Chlorine	}	. . . . .	0.001 per 1000	
Bromine				
Iodine . . . . .			0.005—0.01	„
Hydrochloric acid	}	. . . . .	0.01	„
Sulphurous acid				
Ammonia . . . . .			0.1	„
Sulphuretted hydrogen . . . . .			0.1—0.15	„
Carbonic oxide . . . . .			0.2	„
Carbon bisulphide . . . . .			1—1.2	„
Carbonic acid . . . . .			10—20	„



## CHAPTER VI.

OZONE ( $O_3$ )—AQUEOUS VAPOUR—PEROXIDE OF HYDROGEN.

## OZONE.

THIS gas is an allotropic oxygen, in which the molecule is represented by 3 atoms of oxygen instead of 2. It is a gas with a peculiar phosphorous odour and possessing marked irritating properties upon the mucous membrane of the eyes and nose, and upon the respiratory tract. Schonbein found that a mouse died in five minutes after exposure to an atmosphere highly charged with ozone. The function it performs in nature is to oxidise oxidisable products and thus to purify the atmosphere. It is best prepared artificially by passing electrical discharges through moist air, and hence it will be readily understood that its chief natural source is atmospheric electricity, and that it exists in greatest quantities during and after thunderstorms—when it is also generally associated with nitric and nitrous acids and peroxide of hydrogen. The peroxide of hydrogen is also a powerful oxidising agent, by parting with some of its oxygen and becoming water ( $2H_2O_2 = 2H_2O + O_2$ ); and nitrous acid will also part with its oxygen with great readiness.

According to the late Dr. Tidy:—

1. Most ozone is found after thunderstorms, and least in damp and foggy conditions of the atmosphere.
2. More is found on the coast than inland, especially when sea breezes are blowing.
3. More is found at high than at low levels.
4. More is found in country than in town districts, and

M. Houzeau suggests that this fact may be due to the freer movement of air.

5. More is found in winter (especially after heavy snow-storms) than in summer.

6. More is found during the night than the day, and the greatest quantity at dawn.

7. Western winds in Great Britain contain more than Eastern. M. Houzeau points out that the manifestation of ozone is affected chiefly by the *intensity* of the winds in most cases, except where these blow directly off the ocean.

8. It is rarely, if ever, found in the air of dwelling rooms when occupied.

**Test.**—The *common test* for ozone is that of exposing to the atmosphere a white porous paper (filtering or blotting), after this has been soaked in a solution of potassium iodide and starch, and then dried. Ozone will free the iodine, which then combines with the starch to form the blue iodide, and thus a blue colour is created ( $O_3 + 2KI + H_2O = 2KIO + I_2 + O_2$ ). The papers are exposed in a cage, and it is well to take observations every 12 hours; the cage aids in protecting the papers from direct sunlight, dust and rain, all of which must be excluded, or subsequent fading of the colour ensues. The cage (fig. 44) consists of a double cylinder of very fine wire gauze, and projecting downwards from the upper part of the inner cylinder is a small hook, to which the ozone papers are attached.

The test is a coarse one, however, and fallacy arises from the following causes:—

1. The oxides of nitrogen, peroxide of hydrogen, and chlorine (which may also be present from electrical discharges in the atmosphere), and some volatile organic acids, will produce the same results upon the papers. Sulphurous acid and sulphuretted hydrogen tend to destroy the blue colour.

2. The freed iodine is partially volatilised, and thus its

effect is lost ; while some of it may return to the potash and form inert iodide and iodate.

3. It is impossible to get uniform atmospheric conditions, *i.e.*, the amount of light, moisture, temperature, and wind vary, and make results inconstant ; and the purity and strength of the starch vary.

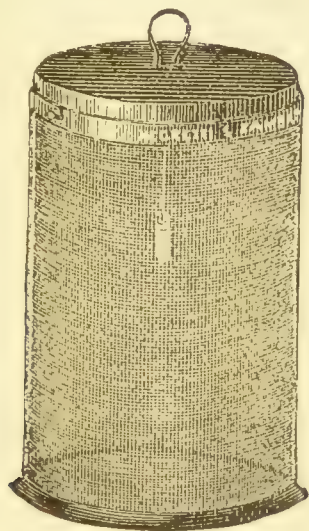


FIG. 44.—The ozone cage.

*A better test* (Houzeau) is the bluing of faintly reddened litmus paper when moistened with a 1 per cent. solution of potassium iodide and dried—when the ozone liberates the iodine and the potash formed gives the paper a blue tint. In the absence of hydrogen peroxide ammonia is the only other gas in the atmosphere which can produce the same effect, and consequently another piece of the litmus paper *not* treated with potassium iodide, is exposed at the same time. Then if the whole colour is not due to ammonia, any difference in the shades of the two papers *must* be furnished by ozone—which can then be estimated by the ozonometer.

The papers must be kept preserved from the air in a tightly closed bottle. In air containing  $\frac{1}{261000}$  of its weight of ozone these papers become blue at once ; hydrogen peroxide

reacts similarly even with these papers, and all ozone estimations are, in consequence, vitiated by this gas. Schöne has pointed out that ozone blackens a bright piece of silver foil, but hydrogen peroxide has no such effect.

The principles upon which a quantitative estimation of ozone (*i.e.*, "ozonometry") is made are colorimetric. The intensity of the blue colour created by the ozone acting upon the papers when these are exposed to the atmosphere, is matched with one of a series of papers forming a standard scale (1 to 10) of tints—each tint having been originally produced by exposing such papers to *known* amounts of ozone. The greater the movement of the air (wind), the greater the quantity which is brought to act upon the paper, and hence less quantities of ozone present on windy days may create more bluing than greater quantities on still days. The only way, therefore, by which an accurate comparison of the ozone in different atmospheres can be made is by lining a dry glass tube with the paper, and aspirating known quantities of the air through the tube.

#### AQUEOUS VAPOUR.

Aqueous vapour must only be briefly considered here.

It is best estimated by hygrometers, *i.e.*, instruments which read the amount of moisture present in the atmosphere. The best of these is probably the combination of wet and dry bulb thermometers, the readings of which when applied to Glaisher's Tables form a ready means of making the calculation.

The wet and dry bulb hygrometer consists of two thermometers, identical in every respect, and fitted on to a stand. The bulb of one of the thermometers (the "wet thermometer") is covered with thin muslin, and around its neck are twisted conducting threads of cotton which pass thence into a small vessel of distilled water—placed at such a distance as to allow a length of about 3 inches of thread; this vessel

is fixed upon one side, so that the evaporation of its contained water may not affect the readings of the dry bulb thermometer. The cotton is previously freed from fat by ether, and then it conducts the water from the vessel to the muslin surrounding the bulb of the thermometer, where it evaporates quickly or slowly, according to the dryness ("drying power") of the atmosphere. As during evaporation there is a reduction of heat, the temperature recorded by the wet bulb is always reduced below that of the dry bulb—except of course in those cases where the atmosphere is already saturated and no evaporation ensues. After the instrument has been exposed for about half an hour, a note is made of the difference in the readings of the two thermometers; from which can be ascertained by means of Glaisher's Tables the various hygrometric data.

The instrument should be placed in the shade, with the bulbs of the two thermometers well exposed—though protected from any radiant heat which may pass from the walls of occupied houses. The observations are generally taken at a height of 4 feet from the ground.

The amount of watery vapour in the atmosphere varies much from time to time and from place to place, and is greatly dependent upon temperature; warm air having the power of holding more invisible moisture than cold. It may range from 30 per cent. of the amount necessary to create complete saturation, to complete saturation itself, taken as 100. From 65-70 per cent. of saturation is probably the degree most conducive to general good health, and 75 per cent. is very agreeable, *i.e.*, a difference between the dry and wet bulb thermometers of about 5 degrees.

Abnormal dryness of the atmosphere gives rise to an oppressive feeling, and obtains chiefly in stove heated rooms, or in rooms ventilated with hot air; and abnormal dampness gives rise to many conditions of ill-health.

All air contains *some* moisture, and when the temperature



is lowered to a less or greater degree, at last—according to the amount of this invisible moisture present—a degree is reached at which the air is no longer capable of holding the vapour and it is deposited in a solid visible form; the temperature to which it is necessary to reduce the air of any place in order that it shall thus deposit its moisture is called “the dew-point.”

The amount of vapour which will saturate a cubic foot of air under the standard barometrical pressure, is given in the following table:—

TABLE II.—Showing the weight in grains of a cubic foot of vapour (*i.e.*, grains of vapour to saturate a cubic foot of dry air), under the pressure of 30 inches of mercury—for every degree of temperature from 32 deg. to 75 deg.

Temperature Fahr.	Weight in Grs. of a Cubic Foot of Vapour.	Temperature Fahr.	Weight in Grs. of a Cubic Foot of Vapour.
Degree.	Grains.	Degree.	Grains.
32	2'13	54	4'71
33	2'21	55	4'87
34	2'30	56	5'04
35	2'39	57	5'21
36	2'48	58	5'39
37	2'57	59	5'58
38	2'66	60	5'77
39	2'76	61	5'97
40	2'86	62	6'17
41	2'97	63	6'38
42	3'08	64	6'59
43	3'20	65	6'81
44	3'32	66	7'04
45	3'44	67	7'27
46	3'56	68	7'51
47	3'69	69	7'76
48	3'82	70	8'01
49	3'96	71	8'27
50	4'10	72	8'54
51	4'24	73	8'82
52	4'39	74	9'10
53	4'55	75	9'39

## PEROXIDE OF HYDROGEN.

This gas is never entirely absent from air, but it exists in greatest quantities during and after thunderstorms. It has been seen that the gas has similar reactions to those of ozone; it may be distinguished, however, by the fact that it is only after the lapse of several hours that it reddens potassium iodide paste. A good test for the presence of hydrogen peroxide is the following:—To some distilled water that has been made to take up the gas add a drop of a 1 per cent. solution of potassium chromate, followed by a little ether and a few drops of dilute sulphuric acid; on shaking, the ether takes up the blue colour of perchromic acid; the test is fairly delicate.

## CHAPTER VII.

## SUSPENDED MATTER IN THE AIR.

THE nature of the suspended matter found in the atmosphere must, of course, vary widely with the place and the circumstances of its collection; and it would not be going too far to say that particles of almost everything the observer can see about him may be present to a greater or less degree. Under these circumstances the task of tabulating these various materials would become a difficult and tedious one; but those which are of more common occurrence are given.

Although under general conditions they are invisible to the unaided eye, everyone must have been struck with the enormous quantities of these suspended matters when a ray of sunshine enters a room and the rays of light reflected from their tiny surfaces disclose their presence. Obviously their number increases according to the extent to which the atmosphere departs from its state of greatest purity; high mountain air on the one hand containing few, and low town air containing many (soot, dust, etc.).

These minute solid particles, which mostly have a tendency to settle when the air is still, gain access to the atmosphere chiefly in the following manners:—

They are lifted up by air currents; by the bursting of the bubbles which may form upon the surface of liquids; by the ascensional force of evaporation; by combustion; and by volcanic upheavals.

It is more especially in factories and workshops that this examination of suspended matters is important, since both

the nature and amount of these have been abundantly proved to be concerned in the production of disease.

The collection and microscopical examination of the dust which settles in a room gives a rough means of investigating the subject—but it is necessary to deal with methods which are more inclusive and precise.

**The methods of collection.**

Undoubtedly the *best method* is to aspirate large volumes of the air slowly through small amounts (say 100 c.c.) of distilled water, placed in 2 or 3 small wash bottles, connected together by tubing. The waters may then be mixed and evaporated to about 50 c.c., when drops may be mounted and examined by the microscope.

The method can be made quantitative by aspirating measured quantities of air through the set of wash-bottles, mixing the waters, and then counting the number of particles in an aliquot part; or the water may be evaporated in a weighed platinum dish, and the weight of residue collected will be the weight of suspended matter in the volume of air aspirated; this may be estimated also as “volatile” and “non-volatile” by noting the loss on ignition.

*Another plan* is by means of *Pouchet's aëroscope*.

This instrument consists of a vertical glass cylinder, capable of being hermetically closed at either end by a copper ferrule. The ferrule at the upper extremity of the cylinder is a permanent fixture, and gives passage to a vertical copper tube which is partly outside and partly enclosed within the cylinder; of this tube the extremity of the part which is outside the cylinder is expanded into a trumpet-shaped mouth, and the end of the part which is inside the cylinder is gradually drawn to a very fine point, not more than 0.5 m.m. in diameter.

The ferrule at the lower extremity can be removed, so that a circular glass plate—which has been previously smeared with pure clean glycerine—can be placed immediately under

the finely drawn point of the copper tube. The whole apparatus is then made air-tight, and connected with the aspirator. The air which is thus sucked in falls in a spray upon the glass slide and the glycerine retains the suspended matter.

A modification of Pouchet's *aëroscope* is the instrument

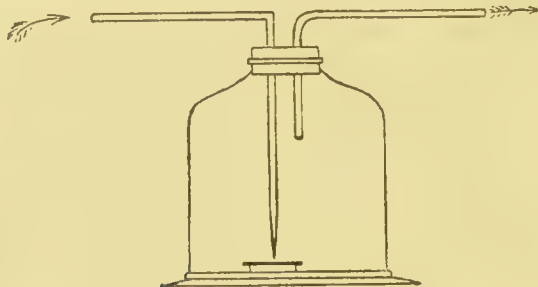


FIG. 45.—M. Marié-Davy's modification of Pouchet's *aëroscope*.

of M. Marié Davy, a description of which is not necessary, for the accompanying figure sufficiently explains itself.

*Hesse's* apparatus is seen by fig. 46 to consist of a long glass tube connected at one end to the aspirator; the small india-rubber cap which closes the other end is removed just before use and 50 c.c. of pure glycerine is poured into the tube, which is then turned round so as to make the glycerine coat the whole interior. As the air is subsequently aspirated through the tube, the suspended matter is caught up by the glycerine—which can be removed and examined microscopically.

Methods in which glycerine is employed have been termed unsatisfactory, for the reason that the original glycerine will generally show marked evidence of solid particles. A preliminary microscopic examination of the glycerine does not, however, entail much additional labour or time.

A *third plan* entails the use of a pure sugar filter through which the air is slowly drawn; the sugar is then dissolved in a sufficiency of pure water and the suspended matters caught



up in it are retained in suspension in the water, when they may be collected and examined microscopically. The filter is best arranged as a glass tube, at least an inch in diameter, disposed horizontally, and packed (not too tightly) for several inches with the sugar crystals. One end of the tube is left

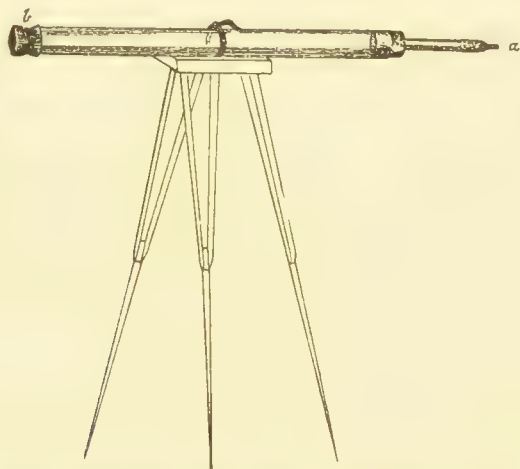


FIG. 46.—Hesse's apparatus for the collection of suspended matters in the air. *a*. The extremity connected with the aspirator. *b*. A removable cap.

open for the entrance of air, and the other connected by india-rubber tubing with an aspirator. The filter dissolved, the suspended matter may be separated by filtration through a weighed Swedish filter-paper, then dried at a low temperature and weighed. If the amount of air aspirated has been recorded, the weighed quantity of its original suspended matter may be expressed as milligrammes per cubic metre, *i.e.*, a quantitative estimation may be made.

Objections may be raised to each of these processes, but such are not of great import; it may be argued for instance—and truly so—that some substances will, in the aspiration method, be deposited in the tubes and bottles. These considerations will not affect the general conclusions arrived at

from such an examination. The dust in town air must obviously vary considerably ; commonly the extent of this variation is between 5 and 25 milligrammes per cubic metre ; it has been estimated as high as 224 in cement works during work.

### THE NATURE OF THE SUSPENDED MATTER.

The more common substances found are :—

#### **Animal.**—

Debris from wear and tear of clothes, etc. ; wool, silk, etc., fibres ; human hair ; particles of feather ; molecular debris (?), in considerable quantity ; debris of dried epithelial cells, and epidermic scales from skin ; pus cells ; fragments of insects, *i.e.*, scales from wings, legs ; particles of the spider's web ; dried fæcal particles from horses' dejecta, etc.

Minute ova ; amœbiform organisms.

#### **Vegetable.**—

Particles of carbonaceous matter ("soot"), etc. ; molecular debris (?) in large quantity ; vegetable fibres, hairs, and cells, etc. ; cotton, etc., fibres ; starch grains ; portions of plants, and pieces of woody fibre ; pulverised straw ; dead spores, etc., of moulds, fungi, diatoms, and bacteria.

Pollen grains ; spores of fungi, moulds, and diatoms (which may even live and grow in the atmosphere), and rarely mycelia of fungi ; algæ, notably *protococcus pluvialis*, and also the small oval cells of other unicellular algæ ; bacteria and their spores. The spores and mycelium of *Achorion Schönleinii* and *Tricophyton tonsurans* have been found in the atmosphere of skin wards.

#### **Mineral.**—

Especially numerous when the ground is dry. Minute particles of every chemical constituent of the soil may be raised up into the atmosphere, *e.g.*, silica, silicate of alumina, salts of sodium, potassium and calcium, peroxide of iron, etc. Sodium chloride is invariably present, and in greatest quan-

tities at the sea-side. Lead, arsenic, and zinc may be furnished by the wall-papers, paint, and "dryers" employed upon the walls of rooms; and arsenic also from artificial fruit, flowers, curtains, etc., used for ornamentation. Coal, dust, etc.

There are certain trade-dusts which vitiate the air of the immediate neighbourhood in which the trade processes are carried on; and particles of the following substances may find their way into the atmosphere:—

Coal and tin, in coal and tin mines; and coal dust also exists to a considerable extent in furnace and engine rooms.

Stone, in quarries, and places where the stones are ground, etc.

Slate, in quarries, and places where slates are prepared.

Cement, in cement works, etc.

Sand, where collected and employed.

Wood, as sawdust in saw-mills, carpenters' shops, etc.

Soot, when chimneys are swept, or when chimneys smoke, etc.

Earthenware, clay, and china—in the work-rooms.

Steel, where such is ground and worked up—as by cutlers, file-makers, steel-grinders, needle and pin makers, tool makers, etc.

Lead, where such is being worked with—as by file-makers, printers, white-lead manufacturers, plumbers, painters, glaziers, lapidaries, lead-miners, type-founders, glazed card manufacturers, earthenware manufacturers, etc.

Fabrics of clothing, *i.e.*, wool, silk, cotton, linen, flax, etc., in factories of these articles.

Wheat and other flour, in mills, bakeries.

Arsenic, in wall paper and artificial flower manufactories.

Copper, in brass foundries, copper smitheries and tin-plate works.

Bichromate of potassium, etc., in manufactories of such.

Pearl-dust, in button, etc., manufactories.

Glass, in glass-works, sand-paper making, etc.

Phosphorus in match manufactories (especially before the red or amorphous phosphorus was used).

Mercury, in silvering and gilding works—before electrolysis was employed.

## CHAPTER VIII.

## THE CHARACTERS OF THE AIR COLLECTED FROM VARIOUS SOURCES.

## MARSH AIR.

THE air collected over marshy regions is contaminated by the products of vegetable putrefaction and fermentation.

*Such air contains:*—Excess of carbonic acid—0.055 per cent. being about the mean; marsh gas in considerable quantity; sulphuretted hydrogen is also sometimes in considerable quantity; watery vapour in large amount; ammonia in traces; ozone in faint traces generally; phosphuretted hydrogen in faint traces. Decaying organic matters (vaporous and solid), together with living minute forms of animal and vegetable life, constitute almost entirely the suspended matter; such is found, therefore, to mainly consist of vegetable debris, algæ, diatoms, fungi, bacteria and other micro-organisms (which in some cases may include the *bacillus malarix*).

In many cases where the presence of sulphuretted hydrogen is marked the marshy waters contain soluble sulphates which become deoxidised to sulphides by reducing agents (chiefly organic matter), and the sulphuretted hydrogen doubtless results from the action of vegetable ("peaty," etc.) acids upon these sulphides.

## SEWER AIR.

Sewer air is of course variable in composition. Its reaction is generally alkaline. The temperature practically never falls below 48 deg.



Oxygen is variously diminished, according to the efficiency of the sewer ventilation; it is sometimes in normal proportions.

Carbonic acid is variously increased from the same cause; it probably does not average more than from 2 to 3 times the normal amount.

Ammonia, markedly increased.

Sulphuretted hydrogen	}	are present in increased quantities.
Ammonium sulphide		
Carbon bisulphide		

Marsh gas is small in amount, or absent.

The foetid and putrid organic vapours of sewage are, according to Odling, allied to the compound ammonias and are probably carbo-ammoniacal, and contain traces of ptomaines and leucomaines (*i.e.*, animal alkaloidal substances).

The micro-organisms are almost exclusively moulds and micrococci, and these, together with animal and vegetable debris, appear to constitute almost the entire suspended matter. Micro-organisms average about 6 per litre in the air of a good sewage system.

The organic matter in sewer air probably averages about three times the amount in the outside air.

#### AIR OVER BURIAL GROUNDS.

The oxygen is slightly diminished.

The carbonic acid varies, but is said to be above the normal.

Ammonia	}	in faint traces.
Ammonium sulphide		
Sulphuretted hydrogen		
Organic vapours.		

Animal and vegetable debris, fungi, bacteria, etc.

## THE AIR OF COAL MINES.

The oxygen is diminished, and the carbonic acid is increased in proportion to the inefficiency of the provisions made for ventilation; marsh gas, in small or large amount; generally a little sulphuretted hydrogen is present; organic matter—chiefly vegetable; few moulds, fungi, or bacteria.

Blasting by gunpowder would add, *inter alia*, carbonic acid, carbonic oxide, and sulphuretted hydrogen to the atmosphere.

## THE AIR DURING FOGS.

Reaction acid; the oxygen is diminished; the carbonic acid very much increased—may even exceed 0.09 per cent.; sulphurous acid and sulphuric acids very much increased; carbon bisulphide in traces; carbonic oxide in traces; ammonia, ammonium sulphide or carbonate in small quantity; sulphuretted hydrogen generally in faint traces; watery vapour excessive; fine suspended particles of carbon and tarry matters, together with the commoner forms of suspended matter in air (*quod vide*), of which there is a considerable increase.

## GROUND AIR.

Ground air may be sucked from considerable distances into a house, especially during periods of frost, owing to the aspirating effect of the warmed and expanded air of the house itself; and the foul air of cesspools has been shown to be sucked through the earth into a dwelling, for distances of over 20 feet. When it is borne in mind that many houses contain cellars built in and ventilated considerably below the ground level, it is not difficult to see how "ground air"

must enter materially into the constitution of the atmosphere of such cellars.

A consideration of the character of ground air will show its undesirability. It contains an enormously high percentage of carbonic acid, and the maximum amount of this im-

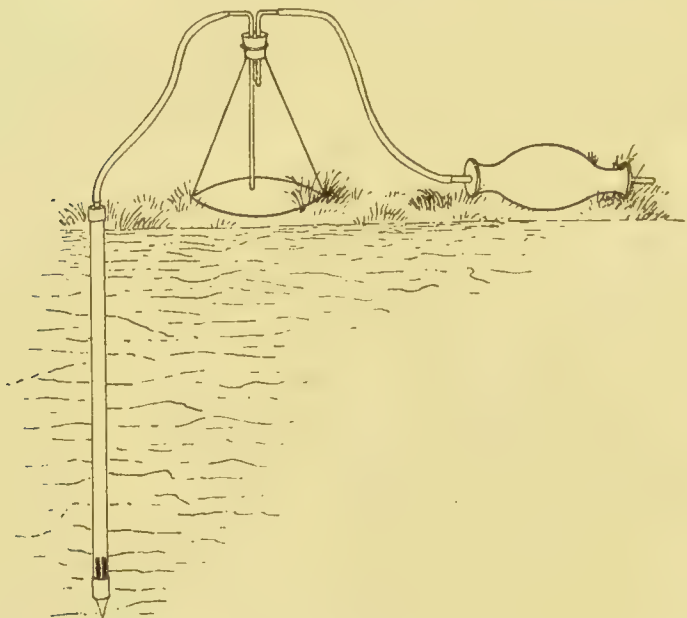


FIG. 47.—Hesse's apparatus for collecting ground air.

purity is always found between July and November—doubtless due to the amount of vegetable life which dies off in the early autumn, and the circumstance that at that season the temperature and moisture prevalent favour its rapid decomposition.

The escape of large quantities of ground air into basements and cellars will be best detected by examining the external atmosphere for carbonic acid, and any considerable excess of this gas not otherwise accounted for points to such pollution; other impurities likewise being present it becomes a question as to where these are derived from, and the matter

can be settled by collecting a sample of the ground air in the vicinity of the house, analysing it, and comparing it with the air of the house basement.

A sample of ground air is conveniently taken in the following way :—A sharp-pointed narrow steel cylinder, with numerous perforations just above its point, is driven into the earth to depths varying from 1 to 6 feet. The upper end of the tube is connected with a large air-jar, which is again connected to an aspirator. The connection between the jar and the steel cylinder is shut off, and the jar is first emptied, by means of the aspirator, of the air it contains ; the connection is then re-established and the sample collected.

Ground air commonly contains a high amount of carbonic acid, and traces of ammonia, sulphuretted hydrogen, and hydro-carbons ; it is comparatively free from micro-organisms.

## CHAPTER IX.

## SCHEME FOR THE DETECTION OF GASES WHEN PRESENT IN LARGE QUANTITIES.

*(Mainly constructed for use of Candidates for Examinations in Hygiene).*

WHEREAS for the detection of the various gases which contaminate an atmosphere, no better course can be pursued than that of passing air through doubly distilled ammonia-free water (or some appropriate absorbing reagent), and then treating the water according to the tests already given; yet, when these gases exist in considerable quantities (as in the atmosphere of chemical manufactories, or those manufactories in which chemicals are employed), they can be discovered by tests applied directly to small quantities of the air itself—such as by the exposure of strips of filtering paper appropriately prepared, etc.

To test the knowledge of candidates presenting themselves for examination in Public Health, generally a considerable amount of a gas is put into a small air-jar; and two or three of these jars, containing each a different gas, are placed before the candidate. In these cases he will do well to adopt the following ready method of procedure which has been devised to meet these special circumstances, and is of course of no use where small quantities of gases are concerned.

1. Partially remove the stopper, smell, and replace as quickly as possible.

Free chlorine has a peculiar and disagreeable odour, and



is particularly irritating to the throat and lungs ; when in very small quantities the odour closely resembles that of seaweed.

Hydrochloric acid has a fainter chlorine-like odour.

Carbonic, nitric, and nitrous acids have no characteristic smell.

Sulphurous acid has a pungent sulphur-like smell.

Ammonia, ammonium sulphide, and sulphuretted hydrogen all possess well-known and characteristic smells—the latter two of rotten egg.

Carbon bisulphide has a peculiar and disagreeable garlicky odour.

2. Moisten two pieces of delicate red and blue litmus papers in neutral distilled water and catch these between the stopper and the neck of the bottle in such a way that they hang down into the bottle free of the sides. Note any change in the colour of these papers after waiting a minute or so.

3. If the reaction is acid or alkaline pour rapidly into the jar a small quantity of distilled ammonia-free water (*i.e.*, about 10 c.c.), and replace the stopper at once ; then shake vigorously several times, so that the bulk of the gases may be taken up in the water.

4. Half of this water is then poured into a test-tube and tested for the gas it contains.

A. *If the blue litmus paper turns red* (*i.e.*, the gas is acid), it is either carbonic acid, hydrochloric acid, sulphurous acid, nitric or nitrous acids.

*Add a drop or two of a solution of silver nitrate* to some of the distilled water emptied from the jar into a test-tube.

(a) *There is a white precipitate*, which denotes the presence of either :—

1. *Carbonic acid*.—*Very* slight precipitate, which is insoluble in nitric acid ; acidity also *very* faint ; clear baryta water added to the jar becomes turbid— which turbidity is increased by adding ammonia.

2. *Hydrochloric acid*.—*Marked* precipitate ; insoluble in nitric acid, but soluble in ammonia, and potassium cyanide ; acidity also *marked*.
3. *Sulphurous acid*.—*Marked* precipitate ; soluble in nitric acid ; the original precipitate on being heated clears up and the solution darkens ( $\text{Ag}_2\text{S}$ ). The water from the jar will decolorise iodide of starch solution, and if it be warmed after the addition of hydrochloric acid and zinc, a piece of lead acetate paper held over the test-tube becomes darkened ( $\text{SO}_2 + 3\text{H}_2 = \text{SH}_2 + 2\text{H}_2\text{O}$ ).

(b) *There is no precipitate*, from which is inferred the presence of either :—

1. *Nitric acid*.—Add brucine and sulphuric acid to some more of the water from the jar, and note the appearances of the pink zone changing to yellow and brown ; or add a crystal of ferrous sulphate and then sulphuric acid to the water, and note the brown coating of the green crystal.
2. *Nitrous acid*.—Add a drop each of the solutions of starch and potassium iodide, and then a drop of sulphuric acid—a blue colour forming at once denotes the presence of this acid ; or the meta-phenylene-diamine test may be applied.

B. *If the red paper is turned blue* (*i.e.*, the gas is alkaline), it is either :—

1. *Ammonia*.—Add a drop or two of Nessler's reagent to a little distilled water from the jar, and a yellow to amber colour appears ; odour characteristic.
2. *Ammonium sulphide*.—Nessler's reagent causes a black colour to appear in the water shaken up in the jar ; nitro-prusside of sodium creates a violet colour ; odour characteristic, *i.e.*, that of rotten eggs predominates, but a deep sniff will always also detect the presence of the ammonia.

C. *If the litmus is not affected (i.e., the gas is apparently neutral), it is either :—*

1. *Sulphuretted hydrogen*.—Lead acetate papers placed in the jar are darkened, as are also solutions of lead, iron, or copper salts ; odour characteristic.
2. *Carbon bisulphide*.—Is a colourless very volatile liquid at ordinary temperatures, with a highly inflammable vapour of a garlicky odour. A drop on a white porcelain slab when lighted burns with a blue flame, giving off sulphur fumes, and leaving a yellow deposit of sulphur behind.

D. *If the litmus papers are first reddened and then slowly bleached, the gas is :—*

1. *Chlorine*.—Filtering paper moistened in a solution of potassium iodide and suspended in the jar is first darkened and then bleached ; odour characteristic ; furnishes a red colour with a mixture of sulphocyanide of potassium and a protosalt of iron.

NOTE.—Sulphuretted hydrogen has many reactions in common with ammonium sulphide, *e.g.*, both gases will darken lead acetate papers, and solutions of lead, copper or iron salts, and their odours are closely similar ; but they may be readily told apart if attention is paid to the subjoined differences :—

*Ammonium sulphide*.—Alkaline reaction ; creates a violet colour with solutions of nitro-prusside of sodium ; odour of rotten eggs and ammonia.

*Sulphuretted hydrogen*.—Neutral reaction ; no effect upon the nitro-prusside ; odour of rotten eggs alone.

## PART IV.

### COAL GAS ANALYSIS.

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#### CHAPTER I.

##### THE COMPOSITION AND ANALYSIS OF COAL GAS.

As it issues from the retort crude coal gas—the volatile product of the destructive distillation of coal—contains large quantities of “impurities” in the form of water, tarry matters, ammonia, carbonic acid, sulphuretted hydrogen and carbon bisulphide; and it is therefore necessary to effect considerable purification. The ammonia has to be removed because it is a diluent, and is likely to give rise to objectionable products; carbonic acid because it reduces the illuminating power of the gas, and sulphuretted hydrogen because its combustion gives origin to sulphurous and sulphuric acids. This purification is done by passing the gas through *condensers*, which remove most of the watery vapour and tarry matters and some of the ammonia (absorbed by the condensed vapour); through *scrubbers*, which remove the remaining ammonia and tarry matters, and most of the sulphuretted hydrogen and carbonic acid; through *lime purifiers*, for the removal of the remaining carbonic acid; and lastly, through purifiers of an iron salt (generally an oxide) for the further abstraction of sulphuretted hydrogen—a sulphide of the metal being formed. Sometimes an additional small purifier of moist calcium sulphide is used to remove the carbon bisulphide that remains in the gas.

Coal gas is thus seen to be a compound gas, consisting of

illuminants, diluents, and impurities; the odoriferous constituents are acetylene, the higher hydrocarbons—such as naphthaline, and organic sulphur compounds.

The illuminants are benzene, propylene, naphthalene, and ethylene. The hydrogen, marsh gas, and carbonic oxide dilute these illuminants, and themselves burn without creating luminosity—though furnishing heat.

The composition of coal gas must vary in every case, of course, according to the efficiency of the means taken to purify the crude gas, but an average of the London companies would be about:—

Hydrogen	...	...	...	50
Marsh gas	...	...	...	32
Carbonic oxide	...	...	...	7
Illuminants	...	...	...	6 (ethylene = 3)
Carbonic acid	...	...	...	1
Nitrogen	...	...	...	2
Sulphurous acid	...	...	}	2
Sulphuretted hydrogen	...	...		
Carbon bisulphide	...	...		
Ammonium sulphide	...	...		

In gas which has been but little purified, traces of many complex substances may be present (mostly alcohols and hydrocarbons).

*After combustion* the following are the average results:—

Nitrogen	...	...	...	...	67
Water	...	...	...	...	16
Carbonic acid	...	...	...	...	7
Carbonic oxide, variable, and least when combustion is most complete; generally about					...
					5 to 6
Ammonia, etc.	...	...	...	}	4
Sulphurous acid	...	...	...		

The sulphurous acid rapidly becomes sulphuric in the atmosphere.



## THE ANALYSIS.

It is beyond our purpose to go into the matter of a complete analysis of coal gas; what concerns us mainly is to learn whether it is sufficiently purified that the products of its combustion shall not yield injurious amounts of harmful gases (such as sulphuretted hydrogen and other sulphur compounds, etc.) to the atmosphere of our rooms. The value of the estimation of the sulphur compounds is of especial hygienic import, and Professor Corfield has pointed out that when coal gas gains admittance to the atmosphere in constant though small quantities—as from defects in pipes or burners—people breathing this atmosphere are apt to contract relaxed and ulcerated conditions of the throat, due in all probability to the sulphur compounds which are added to the atmosphere by the escaping gas.

When the escape is in large quantities into an atmosphere more or less confined the result may be fatal to those breathing it; this fact is due chiefly to the large amount of carbonic oxide which thus gains access to the air, and the symptoms are accordingly those of poisoning by this agent.

When the gas is incompletely burnt (as when defective burners are used), the air of the room becomes disagreeable and somewhat irritating to the throat and larynx. The injurious products of incomplete combustion of coal gas are sulphurous acid, which soon becomes sulphuric in moist air, carbonic oxide, and carbonic acid.

*The Gaseous Compounds of Sulphur.*—The sulphur in coal gas has been estimated as high as 60 grains in 100 cubic feet of gas; but its amount will necessarily vary with the pyrites in the coal employed, the manufacture of the gas, and the efficiency with which provision for purification is made and maintained. The sulphur is mainly in the

form of sulphuretted hydrogen, sulphurous acid, and carbon bisulphide.

*Sulphuretted hydrogen.*—This harmful constituent of crude coal gas (of which it may form a considerable percentage) can be so easily and thoroughly removed by the employment of efficient purifiers, that there is no excuse for its presence in the gas supplied to consumers. By Acts of Parliament all gas supplied must be wholly free from this impurity; a quantitative estimation, therefore, is unnecessary, since any trace suffices for condemnation.

In making a qualitative determination the Gas Referees recommend that the gas should be passed, as it leaves the service pipe, through an apparatus (fig. 48),\* in which are suspended slips of bibulous paper impregnated with the basic acetate of lead (1 part of sugar of lead to 8-9 parts of water). Such papers become darkened in the presence of sulphuretted hydrogen, etc., in degree varying with the amount of this agent present; the faintest indication will generally be found in a slight discoloration of the extreme margin of the papers.

*Sulphurous acid and sulphur compounds other than sulphuretted hydrogen.*—These are estimated by collecting the sulphur as sulphuric acid, into which it is converted by combustion; precipitating this by baric chloride as the sulphate of baryta, and then estimating the sulphur in the precipitate (of  $\text{BaSO}_4$ ).

The gas is passed through a *meter*, by means of which the rate of flow can be adjusted and registered to half a cubic foot per hour, and in which a self-acting movement shuts off the gas when 10 cubic feet have passed through. The meter employed is shown in fig. 49. The dial of the meter is divided into 100 parts, and each complete revolution of the index hand represents that one cubic foot of gas has

\* For the illustrations of apparatus required in gas analysis, I am indebted to Messrs. William Sugg and Co., Gas Engineers, London.

passed through the apparatus ; and each division, therefore, represents  $\frac{1}{100}$  of a cubic foot. The position of the long index hand on the dial must, accordingly, be carefully noted at the commencement of each test.

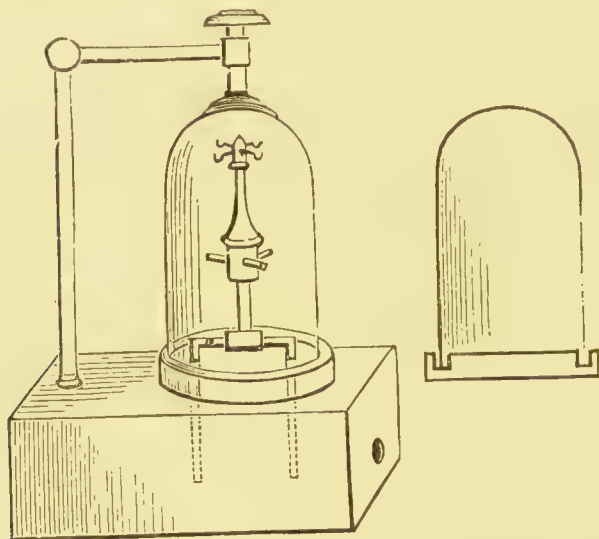


FIG. 48.—Apparatus for testing the presence of sulphuretted hydrogen.

When making the estimation no other gas should be burning in the same room, and, according to the directions of the Referees, the gas (which has been freed from sulphuretted hydrogen) is to be burnt in a small Bunsen burner mounted upon a short cylindrical stand (figs. 50 and 51), which has on its upper surface a deep circular channel to receive the wide end of a trumpet-shaped glass tube. On the top of the stand, between the narrow stem of the burner and the surrounding glass tube, are to be placed pieces of commercial sesqui-carbonate of ammonia, weighing in all about 2 oz.

The products both of the combustion of the gas and of the gradual volatilisation of the ammonia go upwards through the trumpet-tube into a vertical glass cylinder packed with glass balls (to break up the current and promote condensation) ; from the top of this cylinder a long glass pipe issues

and serves to effect some further condensation, as well as to regulate the draught and afford an exit to the non-condensable gases. In the bottom of the cylinder is fixed a small glass

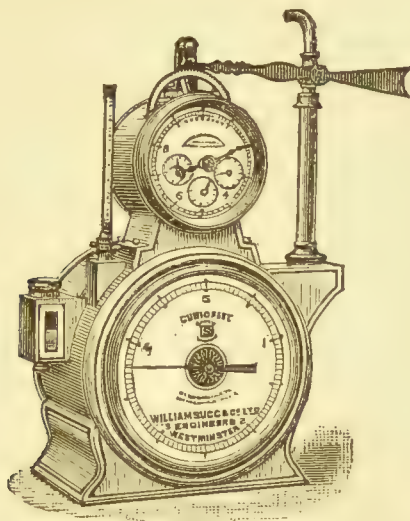


FIG. 49.—The experimental "gas" meter.

tube through which the condensed liquid arising from the combustion of the hydrogen in the gas (and containing the sulphuric acid) passes into a beaker placed beneath.

All parts of the apparatus must of course fit accurately, and the india-rubber connections must permit of no escape. The gas flame, which should not be luminous, is of necessity very low indeed—since only half a cubic foot per hour is burnt; and when 10 cubic feet of gas have passed through, the cylin-



FIG. 50.—Burner with fittings used for estimating sulphur compounds.

der and trumpet-tube are to be well washed with distilled water, and these washings are added to and mixed with

the contents of the beaker. Slight excess of hydrochloric acid followed by an excess of a solution of the chloride of barium are next added to the whole, which is then well boiled for five minutes. The precipitate of sulphate of baryta which has formed must be collected on a Swedish filter paper, and then several quantities of hot distilled water passed through the filter—so that it may be thoroughly freed of any baric chloride and ammonium chloride remaining behind with the baric sulphate. This “washing” is repeated, therefore, until a drop of a solution of

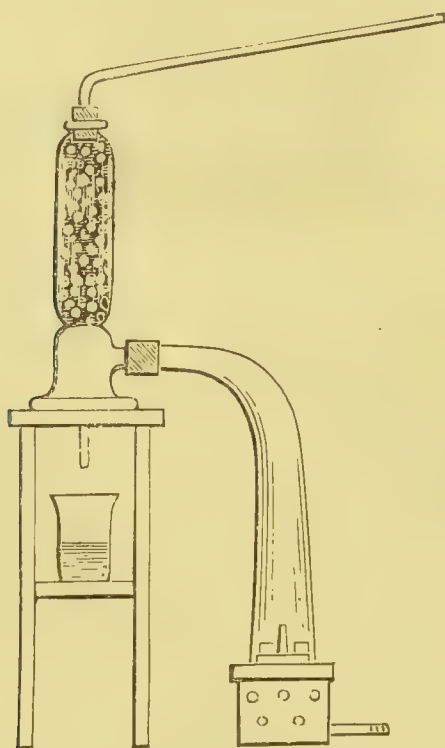


FIG. 51.—Complete apparatus for estimating the sulphur compounds in coal gas.

silver nitrate added to the filtrate in a test-tube gives no cloudiness. The filter paper with its contained baric sulphate has



then to be dried in the water oven; it is next folded up and placed within a weighed platinum dish, which is gradually heated to redness, and retained thus until the filter paper has burnt off. Then the platinum dish is allowed to cool in a desiccator over strong sulphuric acid, and is subsequently weighed. The excess of this weight over that of the empty dish is due to baric sulphate.

∴ Supposing 8.6 grains of baric sulphate are found in 10 cubic feet of gas, then  $8.6 \times 0.137$  will represent 1.178 grains of sulphur in 10 cubic feet, or 11.78 grains in 100 cubic feet.

The number is to be corrected for the variations of temperature and atmospheric pressure, the readings of the thermometer and barometer being taken for the day on which the testing commenced, and also for the day on which it closed, and the mean of the two is taken.

The maximum permissible amount is 17 (22 in the winter) grains of sulphur in every 100 cubic feet of gas on an average of three days (Gas Referees).

*Ammonia.*—Traces of this gas may escape absorption by the water in "the hydraulic main" and the "scrubbers" through which it subsequently passes. This agent, besides adding an impurity to the atmosphere, acts deleteriously upon many of the metallic fittings, etc., about a room. Its presence may be detected by exposing to the gas a filtering paper dipped in the Nessler reagent, and noting any degree of yellowing which may ensue. It is necessary, however, to make a quantitative examination; and the Gas Referees recommend that the coal gas should be passed through a glass cylinder (fig. 52) filled with glass beads which have been moistened with a measured quantity (considerably in excess of any quantity of ammonia likely to be present) of standard sulphuric acid. One terminal tube of the cylinder is connected with the gas supply, and the other with the meter; and the gas is to be passed through at the rate of about half a

cubic foot per hour for 20 hours, so that 10 cubic feet of gas shall be examined. The sulphuric acid fixes the ammonia present, and its acidity is thereby correspondingly decreased. The glass cylinder and its contents are washed out with neutral distilled water, the washings coloured with litmus and tested by a standard solution of ammonia until the colour changes—*i.e.*, until the whole of the sulphuric acid has been neutralised. The difference between the quantity of ammonia solution required to neutralise the quantity of

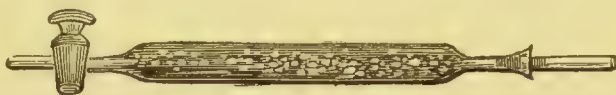


FIG. 52.—Apparatus for estimating ammonia.

standard acid originally employed, and that required by the acid now weakened by the ammonia taken up, represents the amount of ammonia in 10 cubic feet of gas.

The maximum amount of this impurity shall be 4 grains per 100 cubic feet on an average of three days (Gas Referees).

*Carbonic acid* is not considered as an impurity—or rather as an injurious contamination—of coal gas; and since its chief effect, when present in quantity, is to limit the illuminating power of the gas, and this test is specially conducted in all gas examinations, the subject does not here call for further comment.

*Carbonic oxide*.—Every possible effort should be made to rid coal gas as completely as possible from the presence of this very dangerous element, since gas leakages and escapes are of common occurrence in every household. For the methods of making a quantitative and qualitative determination *vide* Air Analysis.

*Tar*.—The gas which issues from the retort is very dark, and contains among its other impurities a considerable percentage of tarry matter; in the process of purification,

however, it becomes quite colourless and all but the very faintest indication of tar is removed. Should the purifying process be imperfect in this respect, and tar be present in even slight amount, a piece of white filtering or blotting paper becomes darkened when held in the gas as it issues from the burner.

## PART V.

## FOOD EXAMINATION.\*

## CHAPTER I.

## COMPOSITION OF COWS' MILK.

MILK consists of water, albuminous substances (casein, albumin, and traces of other kindred nitrogenous substances), milk sugar ( $C_{12}H_{22}O_{11}$ ) and mineral salts (chiefly phosphates of calcium, magnesium, and potassium, and chlorides of sodium and potassium; very small quantities of sulphates are present, and, if any, traces only of carbonates).

The fat is suspended as minute globules, and since this substance forms the lightest element in the milk, after the lapse of time the bulk of it rises to the surface in the form of "cream"—the largest globules being the first to separate. This natural separation of the fat is a slow process, and even after twenty-four hours is not complete; so that given the cream consisted of *pure* fat, it would not represent the total amount of fat which the original milk contained; it is found,

\* Of the samples of food taken and analysed during the past two years under the Sale of Food and Drugs Acts, an average of about 10 per cent. are found to be adulterated; the chief adulteration being practised upon milk, butter, coffee, and alcoholic drinks.

however, that the cream consists—in addition to fat—of varying proportions of water, sugar and casein. The next change the milk undergoes is a souring, followed by a natural separation into a solid (“curd”) and a liquid (“whey”). This change can be readily induced artificially by the addition of calves’ rennet—but the effect is created naturally by the fermentative conversion of the milk sugar into lactic acid, by the agency of a micro-organism which gains access to the milk, and which is known as the bacterium *lactis*.

The “curd” is found to consist of casein, albumin, and traces of other kindred nitrogenous substances; the “whey,” of the water, milk-sugar, and salt.

A still later change is characterised by the appearance of a markedly bluish tint, which is ascribed to the growth of another micro-organism, called *bacillus syncyanus*; and ultimately the casein decomposes owing to the access and development of putrefactive bacteria.

“Koumiss” is a well-known article now upon the market, consisting of mares’ milk which has been skimmed of some of its cream and then partially fermented by yeast—whereby much of the sugar, in which mares’ milk is rich, is converted into lactic acid.

*Cows’ milk* is, of course, the milk which almost entirely concerns the Public Health Analyst; and when the vast amount consumed, the dependence placed upon it as a food article (forming as it does the staple food of childhood), and the peculiar properties and powers which the medium possesses of absorbing and retaining harmful materials and of cultivating micro-organisms are considered, the importance of the subject of milk examination cannot well be over-estimated.

First it is necessary to have a knowledge of the characters of good milk, and a perfect familiarity with this branch of the subject is essential, in order that departures from such may be at once recognised and appreciated.

The average composition of pure cows' milk may be given as:—

Fatty solids	...	...	3.7	} Total solids, 12.6.
Non-fatty organic solids	...	...	8.2	
Ash	...	...	0.7	
Water	...	...	87.4	
<hr/>				
100.0				
<hr/>				

The ash is generally very nearly 8 per cent. of the non-fatty solids.

The cream forms about 10 per cent. by volume, and the specific gravity is generally about 1.032. Milk, however, which has been collected under circumstances which preclude the possibility of any dilution or adulteration has been found to contain these different constituents in amounts varying between considerable limits.

The circumstances which mainly determine the quality of the milk are these:—

(a) The breed of the cow. Alderneys give most fat, and Longhorns most casein.

(b) The time which has elapsed since the last milking.

(c) The stage of milking. That which is first drawn ("fore-milk") contains very little cream (under 0.5 per cent.); but towards the end of the milking the cream is very high in amount; and the very last quantities drawn from the udder, and which are known as "the strippings," are almost pure cream.

(d) The health of the animal.

(e) The age. Young cows secreting less milk—and that of a poorer quality.

(f) The time of year.

(g) The period which has elapsed since last calving—affecting the presence or absence of colostrum corpuscles.

(h) The number of pregnancies—less milk is given with the first calf (Hassall).



(i) The food taken—beet and carrots throw up the sugar.

(j) The conditions under which the animal is kept—house or grass-fed, etc.

What are the limits, then, within which the various components of a *pure* milk may fall?

I am indebted to Dr. James Bell's manual upon "The Chemistry of Foods" for much information under this head. A large number of milk samples were collected from different cows and examined, and the valuable tables compiled afford a trustworthy answer to the above question. These show that the following limits may be reached:—

Fatty solids, 1.92—6.87 per cent. } Total solids, 10.31  
Non-fatty solids, 8—11.27 „ } —16.29 per cent.  
Mineral ash, 0.62—0.87 „

Cream, 2—26 per cent.

Specific gravity, 1026.70—1036.94.

It is very rare, however, that the non-fatty solids fall below 8.5 per cent., or the total solids below 11.5 per cent.

The following table (after Bell) shows a comparison between samples of the milks of various animals.

Constituents.	Cow.	Woman aged 18.	Woman aged 33.	Mare.	Goat.	Ewe.
Specific Gravity . . .	1032.50	1034.50	1033.03	1036.12	1032.70	1039.30
Fat . . . . .	3.76	3.20	2.99	1.76	5.80	11.28
Casein, Albumin, etc.	3.50	2.39	2.51	3.58	4.20	8.83
Sugar . . . . .	4.75	6.83	6.51	5.87	4.94	3.58
Ash . . . . .	0.78	0.29	0.30	0.39	1.00	1.09
Water . . . . .	87.21	87.29	87.69	88.40	84.06	75.22
Total . . . . .	100.00	100.00	100.00	100.00	100.00	100.00

It will be seen from this table how the casein and albu-

min varied in the samples taken of the different milks, from 2.39 in a young woman, to 8.83 in the ewe; with regard to the fat, how this is lowest in the mare's milk (1.76), and highest in the ewe's (11.28); that sugar ranges from 3.58 in the ewe, to 6.83 in the young woman; and that the water is least in amount in the ewe's milk (75.22), and greatest in that of the mare (88.40).

Adopting the cow's milk as a standard for comparison, *human milk* shows an increased quantity of sugar, and a slightly increased quantity of water; but all solid constituents, with the exception of sugar, are materially less. *Mare's milk* is also richer in sugar and water, and slightly so in casein and albumin; the fat and ash are considerably less. *Goat's milk* is richer in all the solid constituents, and of necessity contains a less percentage of water. *Ewe's milk* is characterised by the very high amount of fat, casein, and albumen; the ash is also higher than in cow's milk, but the sugar and water are less.

### THE MILK OF DISEASED COWS.

Although, fortunately, the milk secretion is in abeyance during some diseases, it is not so in all, nor is it even in every case of the same disease; and in a few conditions the milk presents somewhat definite chemical and microscopical characters, which are fairly constant. To the naked eye the milk is commonly all that is desired.

It is in cattle plague and foot and mouth disease that these changes are most marked.

*In cattle plague* the sugar is markedly diminished; the fat is increased, together with—to a less extent—the casein and salts (Gamble). Blood and pus corpuscles are also commonly detected in the milk.

*In foot and mouth disease* the milk commonly contains pus, blood, or mucus (*i.e.*, in those cases where there is ulcera-

tion of the nipples or abscesses in the udder); and, as in cattle plague, the milk corpuscles, under the microscope, are seen to display a tendency to aggregate into grape-like clusters; when the disease is advanced, bodies resembling pus corpuscles (though a little larger), and large yellow granular bodies, together with pus and blood corpuscles, vibriones, and bacteria are also present. In this disease the results of chemical analyses vary so considerably as to be of no value for diagnostic purposes. The milk separates, however, remarkably quickly on the application of a gentle heat, into curds and a pale blue whey; and this feature alone is considered as almost diagnostic by some Continental observers.

In the Western States of America the cows are sometimes seized with an affection commonly termed "the trembles," and this is held to be caused by their feeding upon the herb "Rhus Toxicodendron." If the milk of these cows be given to children, a train of symptoms sets in, *i.e.*, weakness and prostration, a fall in body temperature, vomiting, swelling and dryness of the tongue, and constipation. Boiling the milk invariably destroys these hurtful qualities.

In consumption the milk is not appreciably affected, although it is said to suffer a reduction of fat toward the later stages of the disease. The B. Tuberculosis has been found by several observers in the milk even when there have been no deposits in the udder.

There is no doubt that in some cases the milk of diseased cows has given rise to scarlet fever and diphtheritic throat affection.

## CHAPTER II.

## THE ANALYSIS OF MILK.

**Apparatus used in milk analysis :—**

1. Lactometer.
2. Cream tube.
3. Red and blue litmus papers.
4. Three platinum dishes, and crucible tongs.
5. Chemical balances.
6. Pipette graduated in cubic centimetres.
7. Water-bath and hot-air oven.
8. Soxhlet's apparatus.
9. Graduated cylinders for Schmidt's process.
10. Packet of prepared filtering papers, and fat-free papers for Adam's process.
11. Microscope.

**Reagents :—**

1. Pure ether.
2. Absolute alcohol.
3. Pure hydrochloric acid.
4. Solution of perchloride of iron.
5. Petroleum ether.
6. Turmeric solution.
7. Baric hydrate solution.
8. Dilute sulphuric acid.

**The physical characters :—**

1. *Consistence*.—The milk should be quite opaque when placed in a narrow glass tube; otherwise it has probably

been watered—whereby it acquires a bluish and abnormally transparent character.

Sometimes it is, on the other hand, thick and viscid ; and, on pouring, has an appearance something akin to mucus. Such milk contains large quantities of albumin, and has the property of imparting, when added in small quantities, its own peculiar characteristics to large bulks of good milk. The condition requires elucidation.

“Colostrum”—the milk yielded by recently calved cows during the first few days after the birth of the calf—coagulates much more readily than ordinary milk, owing to the much larger quantity of albumin it contains ; it is also more yellow than ordinary milk, shows flocculi, and has a slightly insipid saline taste. Some milks, however, coagulate immediately after being drawn, or immediately upon warming ; these are, of course, very acid, and are generally yielded by cows in febrile conditions while suffering from inflammation of the udder.

2. *Colour*.—The colour is mostly caused by the minute fatty globules suspended in the fluid and to a slight extent by casein in a very fine state of division ; and everyone has noticed, so soon as the fat is separated in the form of cream, to what a marked extent the original white colour is lost. Normally a good milk should be white with the faintest possible suspicion of yellow—although such food as buttercups, carrots, mangel-wurzel, etc., tend to make the colour distinctly yellow. A distinct yellow may also occur naturally in milk containing considerable quantities of colostrum corpuscles, where the animal is jaundiced, or where there are certain congestive conditions of the udder. This colour is sometimes artificially created by dairymen, by means of the addition of annatto or turmeric.

Rarely, the freshly drawn milk is of a faint blue hue—or even green, or reddish-brown. The cause of these color-

tions has been ascribed to both the food consumed and also to micro-organisms. Certain it is that such milks may be deleterious to health, and have, in fact, been known to cause severe gastro-intestinal irritation; and more especially is this the case with "blue" milk. When these colours form *slowly* after the milk has been passed, it seems probable that they are invariably due to micro-organisms—such as *oidium lactis* or *penicillium*; and in support of this contention Erdmann discovered that vibriones were capable of producing aniline colours from protein substances. The presence of *B. Cyanogenus* may occasion a blue colour in the milk.

A pinkish hue is sometimes created by the presence of blood, but generally the blood shows a tendency to settle.

3. *Taste and odour*.—No taste or odour—other than that of good milk—should be perceptible.

Milk has a marked power of absorbing any odorous gases with which it comes in contact, and of acquiring and retaining distinct flavours from the food consumed by the animal secreting it; thus cows which have been feeding upon turnips, garlic, fennel, etc., yield a milk which tastes of these articles, and when bitter medicines have been administered, or when the chestnut or vine leaves have been eaten, or when the cow suffers from some forms of liver disease, a bitter flavour is imparted to the milk.

4. *Reaction*.—This should be neutral or alkaline; and if acid, only *very* faintly so. Milk is frequently faintly acid by the time it comes to be analysed. If markedly acid, lactic acid fermentation has well set in; and if markedly alkaline, some alkaline salt (such as sodium bicarbonate) has generally been added; or either condition may be due to the fact that the cow is not in health.

5. *Sediment*.—Any insoluble solid matter which may have gained access, and is suspended in the milk, is generally readily seen on the white background which the fluid itself presents; and any sediment is always detected at the bottom



of the cream tube after the milk has stood in this for several hours. Dirt (cow-dung, etc.), pus, blood, epithelium, etc., will deposit on standing.

6. *Temperature*.—The temperature of the milk at the time of performing the analysis should be taken.

A *hygienic analysis* of milk mainly aims at ascertaining whether water has been added, and whether cream has been removed. The addition of such water may also become a weighty consideration to the Health Officer, in those cases where a suspicion of the milk as a probable cause of typhoid, etc., is entertained, for when such milk is found to be watered, a judicious and thorough investigation of the source of the water employed may discover the secret of the milk's infectivity; and the value of the adoption of bacteriological methods, rather than chemical ones, in these cases is apparent.

A hygienic examination of milk, then, must aim at detecting the addition of water or of preservatives and colouring agents, and the removal of cream. It is only in rare cases, *i.e.*, in those where the milk is judged to have incurred special risks, or where the consequences of its consumption point to such contamination, that the common poisonous metals (or other *materies morbi*, such as tyrotoxicon, ptomaines, and bacteria) need be looked for; but it must be borne in mind that milk can absorb metallic poisons (lead, copper, zinc) with great facility when brought in contact with them, that other *materies morbi* (B. Tuberculosis, B. Diphtheriæ, B. Typhosus, etc.) may gain access in a variety of ways, and that milk is a medium which will feed and foster micro-organisms to an exceptional degree.

The milk must be fresh, as after lactic acid fermentation of the milk sugar has set in, there is a slight reduction in the weight of the original solids—non-fat.

In sour milk the fat increases somewhat at the expense of the albuminous material, and the lactic acid, which is soluble

in ether, would be weighed as fat. Sour milk cannot, therefore, be analysed with absolute trustworthiness, and where the acidity is very marked an analysis should not be undertaken.

The sample should in every case be gently shaken before any is removed for analysis. A few drops of formaldehyde will keep a sample fresh for many days.

It will be noted in the analysis that all the various constituents of the milk, with the exception of the cream, are expressed as the percentage they form by weight of the milk—the cream being expressed by its percentage volume.

1. *The cream.*—Some of the milk is poured into a “cream-tube,” which is a glass cylinder; the upper part of the tube bears markings that show the proportion which the cream forms to the total volume of the milk.

The milk is made to stand exactly up to the level of 0 of the scale (due allowance being made for capillarity), and it is then set aside for 12-24 hours. Supposing the cream is found to extend from 0-10 on the scale, then it is 10 per cent.; and if it extends beyond this to the depth say of 2 of the smaller markings between the 10 and the 20, then there is 12 per cent.—and so on.



FIG. 53.  
The cream-  
tube.

The cream will have separated from most milk-samples in 12 hours, but as a rule the separation is not complete for 24 hours; if the time is protracted beyond 24 hours, partial drying ensues, and the resulting contraction will eventually leave a space between the lower surface of the cream and the upper surface of the milk. The average of good milk shows about 10 per cent. of cream, but the milk of Alderney cows frequently yields between 30 and 40 per cent.

The estimation is of considerable value when considered side by side with the specific gravity (*quod vide*), in forming a rapid conclusion as to whether milk

has been tampered with. Unfortunately, however, the whole of the fat does not separate as cream; nor does the amount which separates in different milks form a constant ratio to that which remains behind—some creams being richer and others poorer in fat.

Cream contains about an average of 48 per cent. of fat, and about 40 to 50 per cent. of water; the rest consisting of casein, milk sugar, and ash.

Clotted cream contains about 60 per cent. of fat.

2. *The specific gravity* is taken by a delicate lactometer; the instrument must be *accurate*, perfectly at rest when the reading is taken, and floating quite free from the sides of the vessel containing the milk, and any frothing (from shaking or pouring into the glass cylinder) must be allowed to pass completely off. In taking the height on the stem to which the lactometer floats, it will be seen that, owing to capillarity, the milk immediately around the stem has climbed slightly upward—the extent of this must be allowed for in taking the reading. A correction for temperature is necessary for exact

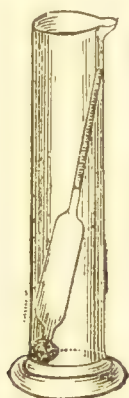


FIG. 54.—Lactometer and glass for taking the specific gravity of milk.

observation, since the instruments are originally graduated by water (taken as 1000) at the temperature of 60 deg. F., and the specific gravity varies with the temperature. The

matter is a simple one, it is only necessary to add or subtract 1 degree of specific gravity for every 10 degrees of temperature registered above or below 60 deg. F.

The specific gravity of distilled water at 60 deg. F. being taken as 1000, that of pure milk at the same temperature is commonly about 1032. This increased specific gravity of milk, as compared with water, is due to the greater amount of solids it contains. The fat is so much lighter than the remainder of the milk, that with its removal an element is lost which partially counteracts the effects of the heavier solids it contains, and therefore the specific gravity rises even higher still. A specific gravity above 1032 will create suspicion, then, as to the removal of the lighter element (cream) from the milk. The dairyman may subvert the detection of this fraud by adding something which will lower the specific gravity again, while at the same time it will increase the volume of the milk. Water is the agent generally employed; it is cheap, always handy (if not required too pure), and it is obvious that the specific gravity of water being 1000, the more of this is added the nearer will the specific gravity of the mixture of milk and water be reduced to 1000. An abundance of cream will naturally account for a low specific gravity of milk; so that a low specific gravity may mean either abundance of cream or the addition of water.

It follows, then, that the two tests considered together must afford a valuable clue as to the class of milk with which we are dealing, at the very outset of our analysis—since a low specific gravity can only be due to abundance of cream in a good milk. If such cream is very low, the sample is either naturally an extremely poor one, or what is more likely, water has been added. If, on the other hand, the specific gravity is exceptionally high, and the cream very low in amount, then there is a great probability that cream has been abstracted.

3. *The total solids and the ash* are estimated in the following manner :—

1. Weigh a small shallow flat-bottomed platinum dish.

2. Charge a 10 c.c. burette with milk, empty into the platinum dish, and then re-weigh, in order to get the weight of milk employed.

3. Evaporate to dryness over the water-bath. It is desirable—and it expedites the process—to break up occasionally, with a piece of platinum wire, the film which forms over the milk and delays evaporation.

4. Complete the drying in the hot-air oven, and remove from time to time, let cool, and weigh, until the weight is found not to vary—at which stage the drying is complete ; or dry for 3 hours and then weigh the solids. The solids must be weighed directly they have cooled, as they are very hygroscopic.

5. The weight found, less that of the dish, will be the weight of total solids in the weight of milk employed.

*Example.*—The platinum dish weighs 12.203 grammes ; the platinum dish + 10 c.c. of milk weighs 22.493.

The weight, therefore, of 10 c.c. of milk = 10.290 grammes (*i.e.*, 22.493 — 12.203).

The platinum dish + total solids = 13.603 grammes.

Deduct the weight of the dish (12.203 grammes), and the difference, *i.e.*, 1.400, represents the total solids.

Then there is 1.400 gramme of total solids in 10.290 grammes of milk. This represents (10.290 : 100 :: 1.400 : *x* per cent.) 13.6 per cent.

6. Ignite the total solid residue until all dark specks, etc., have disappeared, and nothing but a perfectly clean whitish ash remains ; this must be effected slowly, and at as low a temperature as possible—and Dr. Bell recommends that an Argand burner be used in preference to a Bunsen, on this account.



7. The ash is then weighed, and its percentage amount by weight ascertained, as in the case of the total solids. Too large a proportion of ash (that is above 0.75 per cent.) points to the addition of mineral matter—a milk may have, on the other hand, a paucity of ash, due to the copious admixture of water.

8. A rough analysis of the ash is sometimes desirable. It must then be dissolved and tested quantitatively for chlorides, sulphates, phosphates, lime, etc., on the principles detailed under water analysis. Effervescence on the addition of hydrochloric acid denotes adulteration by a carbonate, which will generally be sodium carbonate; the ash of a pure milk does not effervesce when hydrochloric acid is added to it.

The poisonous metals may be taken up, but since milk is only in contact with these for such short periods under ordinary circumstances, it will not be necessary to test for them except in the event of the milk having been boiled in metallic receptacles, or where there are other *special* reasons for suspecting their presence.

9. *Solids—fat*.—The estimation of the amount of fat in milk is, at the same time, the most important and the most difficult step in the whole analysis.

There are many methods at the present day in use for its extraction and estimation. With care Bell's process gives good results, but the fat is thereby slightly underestimated.

**The process is as follows:—**

1. A shallow platinum dish of about three inches diameter is weighed.

2. And into it are placed 10 c.c. of milk.

3. The whole is reweighed so as to learn the weight of 10 c.c. of milk.

4. Place on a water-bath, stirring every now and then with a small clean glass rod (which is left in the dish), until the residue attains the consistence of a tenacious pulp.



5. The solid residue should be moistened, and worked up by the glass rod, with a little absolute alcohol in order to disintegrate it and to assist the ether in extracting it.

6. Half fill the platinum dish with ether, and *thoroughly* mix and pound the residue, by means of the glass rod, so that the ether shall be brought everywhere in contact with the fat which the residue contains.

The ether will dissolve out this fat, and this it does more readily and effectually if it be made hot. A naked flame must not be brought near to the ether, so it becomes necessary, in order to effect the warming, to float the dish and its contents in a little water—heated to the temperature which the fingers can just tolerate for a moment—until the ether boils gently; then remove, and pour off the supernatant ethereal solution of fat on to a small fat free filter paper, and collect the filtrate in a weighed platinum dish.

7. Repeat this treatment by boiling ether three times; or if the residue by then has not acquired a thorough whiteness, or has not been thoroughly broken up into a fine sediment—a fourth time. It is never necessary to proceed beyond a fourth treatment if each has been performed thoroughly.

8. Cut off the upper part of the filter and place it with the lower part low down in the glass funnel, then thoroughly wash with ether so as to remove any fat which may have hung about the paper.

9. Boil away the ether over hot water, and when ebullition ceases transfer to hot-air oven, heated to about 212 deg. F., in order to thoroughly drive off any alcohol, etc.; the small oil globules run together, and nothing but a residue of fat remains behind in the dish.

10. The weight, less the weight of the platinum dish, equals the weight of fat in the weight of milk experimented on.

*Example.*—The weight of the 10 c.c. of milk is found to be 10.202 grammes.

The weight of the platinum dish for receiving filtrate = 11.009 grammes.

The weight of the platinum dish + fat = 11.349; therefore the weight of fat in 10.202 grammes of milk =  $(11.349 - 11.009) = 0.34$  gramme.

$10.202 : 100 :: 0.34 : x$  per cent. = 3.3 per cent. of fat.

11. The solids—non-fat, may be estimated from the residue remaining in the dish from which the fat has been extracted. This is placed on the water-bath until apparently dry, and then removed to a hot-air oven kept at 212 deg. F. When the weight is found to be constant, the weight of the dish plus its contents, minus the weight of the dish, will represent the solids—non-fat. This method of estimating the solids—non-fat, is more exact than that of the more ready method of subtracting the fat from the total solids.

Hehner and Richmond have devised a formula by means of which the fat in ordinary milk samples may be estimated.  $F = 0.859T - 0.2186G$ , where  $F$  = fat,  $T$  = total solids, and  $G$  = the last two units of the S.G. and any decimal (*i.e.*, if S.G. = 1.0295,  $G = 29.5$ ).

#### ADAM'S PROCESS.

This process is now largely employed in this country; it usually gives results showing the fat to be about 0.2 per cent. higher than Bell's process, and the difference must be due to the less complete abstraction of the fat by the latter process.

1. Shake the sample and pipette 5 c.c. into a small beaker about 2 inches deep and  $1\frac{1}{2}$  inches wide; then weigh.

2. Soak up as much of the milk as possible—and in every case almost the entire 5 c.c.—by a coil of white demi-blotting-paper, keeping the beaker covered during the absorption.

It is, of course, imperative that the paper should be freed from fat prior to its employment. This may be done by

extracting the coils with acid alcohol (alcohol containing 10 per cent. of acetic acid) for at least three hours, and then thoroughly drying; or the specially prepared slips of fat-free paper made by Messrs. Schleicher and Schüll, of Düren, may be used. A helical coil is prepared by rolling a strip of the paper  $2\frac{1}{2}$  inches wide and 22 inches long upon a glass rod of the size of a cedar pencil, care being taken not to tear the paper; and the coil may be held together with platinum wire.

3. Remove the coil by its upper part, and place it dry end downwards upon a slip of glass, and then re-weigh the beaker with the trace of milk left behind in it. The difference in weight from the previous weighing represents the weight of milk experimented upon.

4. Dry the coil in the water oven for two hours and then extract the fat by anhydrous ether in a Soxhlet, 12 siphonings at least being necessary.

5. Receive the fat and ether in a small light flask; boil off the ether; dry to constancy in a water oven at about 220 deg. F., the flask being laid in a horizontal position; let cool and weigh. The solids non-fat may be estimated by the difference.

#### NOTES UPON THE PROCESS.

By the addition of ammonia sour milk is as easily taken up as fresh.

To obviate the two weighings some analysts apply the whole 5 c.c. of milk, with suitable precautions, directly to the paper, as by spotting it upon the paper. The tared flask of the Soxhlet should have a capacity of about 150 c.c., and contain about 75 c.c. of ether.

It is well to place a small plug of blotting-paper in the mouth of the open tube at the top of the condenser so as to limit the access of air, the moisture of which would condense and slightly wet the ether.

Soxhlet's apparatus is shown in fig. 55. A is a small flask which has been thoroughly dried and weighed and then about half filled with ether; the extraction apparatus is shown attached to the flask below, and to the condenser (K) above, the latter being fixed in a very slanting position. F repre-

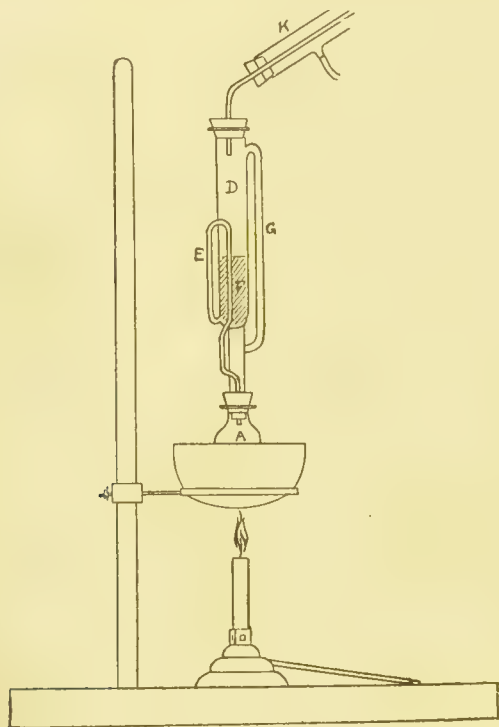


FIG. 55.—Soxhlet's fat-extraction apparatus.

sents a thimble of fat-freed paper (as made by Messrs. Schleicher and Schüll, Düren), the substance to be extracted is placed in this, the capsule is closed by folding inwards its upper rim, and then placed in D, care being taken that it is entirely below the level of the siphon E, so that it may be completely immersed in the solvent, and also that it does not close the opening to the siphon.

The dish on which the flask stands is partially filled with

water, and this is cautiously heated; the ether vapour then ascends G, and passing into the condenser, is at once condensed and drops on to the F; the ether goes on accumulating, rising the while in the ascending arm of E, until it reaches the level of the upper bend, and overflows, when siphonage takes place, and the ether is sucked out of D, and returns to the flask. Thus the circulation of the ether is completed every few minutes, and the substance is repeatedly exposed to the action of the ether for from 3—6 hours, according as it is poor or rich in fat; the flask is then removed, the ether driven off over the water-bath at a temperature sufficient to make the ether boil, and the flask and its contents dried at 212 deg. F., until a constant weight is obtained.

Of course there must be no doubt as to whether the extraction has been complete; this may be tested by fixing a second small flask containing more ether, and after about half an hour evaporating off the ether and drying at 212 deg. F., and noting whether there is any material increase over the original weight of the flask.

#### SCHMIDT'S PROCESS.

This process has become a favourite one, for by it a very accurate estimation of the fat can be made in a very short space of time.

The process as modified by Stokes is as follows:—

1. A graduated tube is employed to receive 10 c.c. of milk, to which 10 c.c. of strong hydrochloric acid is added, the milk and acid thus standing to the mark of 20 c.c. on the tube.

2. The mixture is boiled, with frequent shaking, until it turns a brown colour (from the conversion of the sugar into maltose).

3. Let stand for about three minutes, then cool by partial immersion in a stream of running water.

4. Fill up to the 50 c.c. mark with ether; cork the tube,



and shake for half a minute, and then set aside for five minutes, when the ether will have separated.

5. Accurately pipette off 20 c.c. of the clear supernatant ethereal solution of fat into a tared dish, evaporate off the ether, dry in air-bath at 212 deg. F., and weigh the residual fat.

6. Next notice how many c.c. of ethereal solution remain in the tube, then from the fat estimated in the 20 c.c. calculate the amount of fat in the whole layer of ether.

*Example.*—10 c.c. of milk, with a S.G. of 1031 and 12 per cent. total solids, gives in 20 c.c. ethereal solution 0.227 gramme of fat.

In the tube there remained 6.5 c.c. of ethereal solution—making a total of 26.5 c.c.  $\therefore \frac{26.5 \times 0.227}{20} = 0.367$  gramme of fat in the 10 c.c. of milk, or 3.67 grammes in 100 c.c. But 100 c.c. of milk with a specific gravity of 1031 weighs 103.1 grammes.  $\therefore$  there are 3.67 grammes of fat in 103.1 grammes of milk, or 3.559 per cent. (Calculated from the formula the fat should be 3.53 per cent.)

#### NOTES UPON THE PROCESS.

There floats between the brown mixture of HCl and milk and the ethereal solution, a fluffy stratum of casein; three-fourths of this stratum should be taken as ether in reading off the quantity of the latter.

The acid and milk should not be boiled together for more than two minutes, or the ether takes up a caramel-like substance.

The whole process does not take more than 20 minutes, and it is well adapted for use where the milk has decomposed. The ether employed should be washed.

An instrument termed the *Lactoscope* forms a ready, but rough means of estimating the approximate amount of fat



which the milk contains. The principle of application of all lactoscopic tests is this:—The greater the amount of fat present in the milk the more opaque the medium, and *vice versa*. Into an accurately graduated cylindrical tube of clear colourless glass a little milk is placed; to this water is added, until certain black lines placed upon a vertical porcelain stem which rises up in the middle of the cylindrical tube can be first discerned through the milk. The level of the milk and water is then ascertained by the graduation on the tube, and each such level has been found, by experiment, to correspond to a certain percentage of fat in the milk.

The method is a coarse one, but it serves to furnish a clue as to those samples which more especially require analysis.

#### MICROSCOPIC EXAMINATION.

Good milk under the microscope consists of a collection of round highly refractile oil globules—all of about the same dimensions, with an occasional epithelial cell; and, from 3 to 8 days after calving, colostrum corpuscles are also present in larger or smaller quantities (fig. 56). These latter mostly consist of large yellow cells containing large and small fat globules in their interior, and distinguishable with difficulty from some pus corpuscles which become swollen by the imbibition of milk. Where, however, the animal is not in

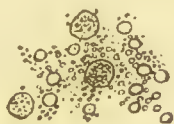


FIG. 56.—Milk. Showing the large colostrum corpuscles. ( $\times 250$ .)

health, the following abnormal constituents may also be found:—

Cast of the lacteal tubes, blood corpuscles (which closely resemble those of the human subject), pus corpuscles, and

various micro-organisms (*e.g.*, fungi—such as *oidium lactis*, moulds—such as *penicillium*, and bacteria). Blood may be detected either by the spectroscope or microscope, or by its chemical reactions; an excellent chemical test is the addition of a few drops of the freshly-prepared tincture of guaiacum and a little ethereal solution of peroxide of hydrogen to the suspected liquid, when a blue colour is furnished if blood is present. When present in considerable quantities blood tinges the milk, and has a tendency to settle.

Cow-dung shows vegetable parenchyma and vessels with a distinct yellow tint.

## CHAPTER III.

## ADULTERATION.

MILK affords an excellent example of each of the various motives for adulteration, viz. :—1. To increase the bulk and weight of the article—in this case chiefly by the addition of water. 2. To disguise the additions thus made, and to give the substance a false strength and improve its appearance—in this case by annatto, turmeric, etc. 3. To preserve the article—here chiefly to prevent its turning sour, by the addition of boracic acid, and rarely salicylic acid and carbonate of soda.

How shall we decide when water has been added, having consideration for the very great variations which have been found to exist in the strength of milks which have not been tampered with? The question is one which has given rise to a great deal of thought and discussion among analysts, but the difficulties are so great that no altogether satisfactory solution has been found; it is clear that the percentage amount of solids—fatty and non-fatty, will be reduced by such addition of water (though these will retain their normal proportions to each other), but then the sample may be naturally poor in solids. The whole question as to whether a sample is a naturally poor one, or has been made so by the addition of water—important as it is, since unscrupulous dairymen may always considerably water a naturally rich milk and yet not make the sample a poorer one than is sometimes naturally yielded by certain cows—is frequently a difficult matter to decide upon.

The estimation of the amount of added water is made from the non-fatty solids, because the amount of these does not fall (in different samples) so far below the average as does that of the fatty solids; otherwise, there are strong reasons for preferring an estimation based upon the fatty solids. The calculation is an easy one. Supposing for instance a sample yields 8 per cent. of non-fatty solids. The low limit of a pure milk which is agreed upon by public analysts is 8.5 per cent., and we are dealing fairly with the vendor by adopting such a limit in all cases.

Then if 8.5 per cent. of non-fatty solids denotes 100 per cent. of pure milk, what percentage of pure milk will 8 per cent. denote?

$8.5 : 8 :: 100 \text{ per cent.} : x \text{ per cent.} = \text{about } 94 \text{ per cent.}$

Therefore, there is about 94 per cent. of *pure* milk in the sample, and  $(100 - 94 = )$  6 per cent. of water has been added.

Wanklyn is favourable to taking the amount of ash as a clue to the estimation of the amount of added water, 0.73 per cent. being taken by him as the limit. His standard, however, is probably too high to exclude the risk of injustice being done to the salesman; moreover, there is considerably more scope for error and variation in the results of different operators when estimating an ash, than exists in the preferable method of making an estimation of the non-fatty solids. It should be remembered that the ash should in every case be low when the solids non-fat are low, or some mineral adulterant has been added.

Next as to the question of *cream-abstraction*. This of course can only be deduced from the amount of fat present; and the difficulty which meets us at the outset is to fix a fair limit of the amount of fat which every sample should contain. This matter has also been much discussed, and has been finally settled by the Society of Public Analysts as 3 per cent.,

but the limit fixed by the authorities at Somerset House is 2·75 per cent. The limit of the percentage amount of fat which must be insisted upon must of necessity be a rather low one, in order to include those samples which are naturally poor; and, in consequence, it is difficult to foresee any measure which shall preclude unprincipled dairymen from diluting rich milk down to this percentage. It is doubtful, therefore, having in view this point, whether it is wise to decide upon a minimum standard of strength, but whether the local circumstances of the breed, food, condition, etc., of the cows should not in every case be used in some measure as a check upon the quality of the milk, consideration being had of the fact that though the milk from the same cow may vary somewhat at times, the mixed products of many animals (dairy samples) vary very little. Dr. Wallace of Glasgow suggests, in this connection, that in every case where the quality falls below a certain standard, the milkman should have the privilege of proving his innocence by having the cow, or cows, milked in the presence of the Inspector or Analyst; and in the case of a man having, say, a dozen cows, it should be no defence for him to show that one of these gives milk of unusually low quality.

The limit of 3 per cent. of fat which has been adopted by the Society of Public Analysts is certainly one which is reached by all genuine dairy samples, but as Bell and others have shown, the milk of individual cows may fall a little below this limit.

The amount abstracted by the removal of cream becomes an easy calculation after an estimation of the fat has been made. Suppose that the solids fat have been found to amount to just 2·5 per cent. Then 3 per cent.—2·5 per cent. = 0·5 per cent. of fat at least must have been abstracted; or  $\frac{2\cdot5 \times 100}{3} = 83\cdot3$  per cent. of the original fat remains and  $(100 - 83\cdot3) =$

16.7 per cent. of the total fat originally in the milk has been removed. In cases where the fat is low and the solids non-fat are high there can be little doubt but that the fat has been abstracted.

It would seem only just, however, to the consumer, to take action in those cases where the fatty solids fall below 3 per cent., and the non-fatty solids below 8.5. If these low limits are not reached it should be no defence to state that the sample was a naturally poor one; it is the average sample that is demanded and paid for by the purchaser, and which he assumes is given him, and anything short of this is not really of "the nature, substance, and quality demanded."

In Berlin the limit of fat is 2.7 per cent., and in New York 3 per cent.

It has been suggested that two standards of quality should be adopted for all milk—a low and a high one; but the objections to such an adoption are seen, from what has already been said, to lie upon the surface, and to make it highly undesirable—since it would sanction the reduction of good milk to the lower standard, by the addition of water or the removal of cream, both of which are offences against the Sale of Food and Drugs Act, 1875.

As milk stands, a certain proportion of the fat rises to the upper layers before it separates so completely as to form a layer of cream, and a defence is sometimes set up by the dairyman that a poor sample was due to the fact that such upper milk had all been removed, and that the sample was some of the last of the milk in the can. This defence is, in most cases, a well recognised subterfuge; and it is, moreover, the duty of the vendor to rouse the milk, and to supply fair samples to one and all alike.

Samples collected on Sunday mornings are generally amongst the poorest, for great inducement is offered to adulteration on these days in order to meet the exceptional demand



—due to the fact that the working class more generally indulge in extra milk upon that day.

In addition to water, there are other adulterants added to milk. Common salt, sodium carbonate, chalk, cane sugar, starch, glycerine and treacle have been used, but they are very rarely, if ever, employed at the present day, and they are comparatively unimportant hygienically.

Boracic, benzoic, salicylic acids and “formaline” are sometimes used as milk preservatives. “Formaline” (containing about 40 per cent. of formaldehyde) may be detected in small quantities by its property of furnishing a blue colour to milk to which sulphuric acid has been added (Hehner).

*Boracic acid.*—Evaporate 100 c.c. of the milk to dryness after rendering alkaline with caustic soda solution; incinerate; extract the ash with a little water rendered faintly acid with hydrochloric acid, filter, and evaporate the filtrate to a small bulk in a porcelain capsule; add a few drops of a fresh saturated turmeric solution and evaporate to dryness. The dried residue is brownish-red, and a transient blue colour results, changing to a green, when a little water and alkali are added to the residue. Or after adding just sufficient hydrochloric acid to the aqueous extract of the ash to furnish slight acidity, dip in a piece of turmeric paper, dry this at a gentle heat, and it turns a brownish-red colour, changing to a dark bluish-green on moistening with an alkali.

A quantitative estimation may be made by distilling it from a sulphuric acid solution with methyl-alcohol, and fixing the boracic acid in the distillate by a weighed quantity of caustic lime (or better, phosphate of soda [Hehner]), contained in a weighed platinum dish, and igniting the residue obtained by evaporation over the blowpipe (*vide* Gooch’s method in text-books of Analytical Chemistry).

Corroborative evidence of the presence of the acid may be got by the flame reaction from the ash, *vide* page 164.

Boracic acid or borax is largely added to milk during the summer months.

*Salicylic acid.*—Slightly acidulate 100 c.c. of the milk with hydrochloric acid, and then thoroughly shake up with half of its volume of a mixture of equal parts of ether and petroleum ether in a separator; draw off the ether, filter, and evaporate; dissolve the residue in a small measured quantity of distilled water, and add a drop or two of a very dilute neutral solution of ferric chloride—a purple colour results in the presence of salicylic acid, which may be titrated by a weak standard solution of pure salicylic acid, and thus an approximate quantitative determination arrived at.

*Benzoic acid.*—Render 250 c.c. of the milk alkaline with baric hydrate; evaporate to about 50 c.c.; make into a paste with calcium sulphate, and dry over the water-bath. Grind to a fine powder; moisten with a little dilute sulphuric acid, and exhaust three times with plenty of 50 per cent. alcohol. The alcohol (which has taken up any benzoic acid present) is neutralised with baric hydrate and evaporated to a small bulk, and this is acidified with weak sulphuric acid and extracted with ether. The ethereal extract leaves, at the ordinary temperature, a residue of almost pure benzoic acid, which is recognised by its aromatic odour and by its volatility; and if the residue is dissolved in water with a little dilute solution of ferric chloride a fine reddish-yellow colour results.

Certainly the addition of preservatives to milk is a grave offence, forming as it does the staple article of food for the susceptible infant population. Such agents may do harm directly, and certainly do harm indirectly by permitting other than fresh milk to be used. Boracic acid, the chief preservative agent employed, has been shown by continental observers to deteriorate the blood corpuscles.

*Annatto and turmeric* are yellow colouring agents, added to give the milk a rich yellow appearance. Potassic hydrate

solution will give a brown colour if turmeric be present ; and if the milk be made slightly alkaline with sodium carbonate and a piece of white filter paper be left immersed in the milk over night, the paper acquires a distinct reddish-yellow tint if annatto is present. Coal tar colours have been employed (*vide* Butter). In these cases, if ammonia be added to the milk, it will, after many hours, impart a yellow dye to a piece of white wool.

**Skimmed milk** is sometimes made to look like good rich milk by the addition of "*condensed milk*." An analysis of the ash and non-fatty solids will, however, detect the fraud, since these will both be in excess of their general proportions, more especially the sugar, and the amount of soluble albumin will be diminished (Faber).

Skimmed milk is generally slightly acid, and the specific gravity is above 1032.5.

The bulk of the samples of "separated milk" contain less than 0.3 per cent. of fat ; it may vary between 0.2 and 0.6 per cent.

## CHAPTER IV.

## BUTTER—CHEESE—TYROTOXICON.

A FRAUDULENT attempt is sometimes made to sell such compounds as "oleo-margarine" for pure butter. In the manufacture of oleo-margarine animal and vegetable fats are melted, strained, cooled with ice, worked up with milk, coloured with annatto, and salted; the result is an article very similar in appearance and taste to ordinary butter, and but little inferior in nutritive qualities; it constitutes a good article of food—but it should not be sold as butter; the only respects in which it is inferior, hygienically, to butter, are that it is less digestible and much more generally contains unwholesome ingredients.

It is opportune, before commencing the method of analysis, to first consider the composition of *pure* butter.

An average sample of fresh butter has the following composition:—

Fat, 83.5 per cent.

Curd (casein), 1 per cent.

Ash, 1.5 per cent.

Milk sugar, 1 per cent.

Water, 13 per cent.

Dr. Bell found, in a great number of genuine samples which he analysed, that the butter-fat may fall as low as 70 per cent., the salts to 0.4, and that the water may vary from 5 to 20 per cent.

The butter-fat is a combination of glycerol with certain fatty acids, and consists of:—

(a) The glycerides of certain fatty acids, *soluble* in hot water, *i.e.*, principally butyric, but also smaller quantities of caproic, capric, and caprylic acids.

(b) The glycerides of certain fatty acids *insoluble* in hot water, *i.e.*, palmitic, stearic, oleic and myristic acids.

But it is believed that the composition of butter is even more complicated, and that the glycerides contain several acid radicles in the same molecule.

The same difficulties are met in agreeing upon a standard of quality to which all butters should be made to conform as in the case of milk, since causes similar to those which effect a considerable difference of quality in genuine milk samples also affect the butter formed from such milks.

It is generally agreed among public analysts that 16 per cent. is a fair limit of water, and that the butter-fat should amount to 80 per cent.

#### THE ANALYSIS.

It is important to note that, after a sample of butter has been seized, it is desirable to proceed with the analysis of it without material delay; and the butter should, moreover, be kept in a cool dark place in the interim of its seizure and analysis, for so soon as it commences to undergo change (decomposition), many of the characteristics which distinguish true butter-fat from other fats tend to disappear to some extent.

If a sample of butter is excluded from the air it may be reliably analysed after many weeks; otherwise it becomes rancid, the insoluble fatty acids tend to increase, and the soluble fatty acids to diminish.

The utilisation of other fats in the manufacture of a spurious butter has called for special legislation; the designation of such compounds was originally "butterine," but this term has now been prohibited by law (1887) as one liable to mis-

lead the more ignorant section of the community into the belief that they were buying genuine butter. All foreign fats, therefore, made up to resemble butter, have now to be labelled and sold as "margarine."

### **Physical Characters :—**

These are too well-known to need detailing here; the smell and taste of good butter are familiar to everyone, and are so characteristic that they form in themselves valuable evidence of its purity. If butter is heated to 70 deg. F., any unpleasant taste becomes more apparent.

With regard to the colour, the same remarks given in connection with the colour of milk apply in a measure to the butter made from it; mention should, however, be made of the fact that annatto is here even more commonly employed as a colouring agent.

We have seen that the main issue of **the analysis** is to determine whether the sample consists of pure butter-fat, an admixture of this with other fats, or of these prepared foreign fats alone. The lines upon which we must proceed to detect fraud must obviously be those which take advantage of any differences existing in the composition of the two kinds of fat; these differences are mainly the following:—

#### **BUTTER-FAT.**

1. The specific gravity at 100 deg. F. is extremely rarely below 910, and never below 909·8.

2. The soluble volatile fatty acids form between 6 and 7 per cent. on an average, and are never below 4·5.

The insoluble fatty acids form about 88 per cent. of its total weight.

#### **THE OTHER FATS MOST USED AS SUBSTITUTES.**

1. Is never above 904 in margarine from beef or mutton fat, but is higher where vegetable fats are employed.

2. Constitute rarely more than half per cent., and never more than three-fourths per cent., in margarine from animal fats.

Generally about 95 per cent.



3. The number of milligrammes of KHO required to saponify 1 gramme (Kœttstorfer's process) =  $221\cdot4 - 232\cdot4$ .

4. Hübl's iodine absorption test gives an iodine deg. of  $23\cdot5 - 39$ .

5. The Reichert-Meissl value (of 5 grammes) is from about  $24 - 34$ .

6. By the Valenta test the fat clears at  $86 - 102$  deg. F.

7. The melting point of the fat varies from  $86$  to  $94$  deg. F., and is commonly from  $88$  to  $91$  deg. F.

8. Readily and completely soluble in ether.

9. Under the microscope pure butter consists of a collection of small oil globules with an occasional large one, and no crystals except when the fat has been melted.

3. Never above  $198$ .

4. Not below  $95$ , so far as vegetable oils in common use are concerned, and rarely below  $50$  in any of the animal fats.

5. The other fats possess a value below  $3$ .

6. No animal fats clear below  $201$  deg. F., and no vegetable oil of common use clears below  $176$  deg. F.

7. Rarely, if ever, above  $82$  deg. F. in margarine from beef and mutton fats.

8. Frequently less so, and quickly deposits a residue, in margarine from animal fats.

9. In margarine from animal fats the contours of the small oil globules are less distinct, and the larger ones are more numerous and irregular in size. Often acicular and stellate crystals of the non-volatile acids are seen.

*The specific gravity test* sometimes suffices for the purpose of detecting in fresh samples fraudulent employment of other fats; it is effected in the following manner:—

1. A quantity of the butter is heated to, and maintained at, near  $150$  deg. F. in a water-bath—made by standing a small beaker containing the butter in a large beaker containing water.

2. The fat slowly separates—and forms an upper stratum,

which rests upon a lower stratum of the water, curd, and salt.

3. In the course of time the upper layer of butter-fat gets clearer and clearer, until at last all the water, curd, and salt having separated, it becomes perfectly clear and transparent. Immediately this has taken place the fat is decanted on to a fine dry filter, in order to guard against the presence of traces of curd and salt, and the filtrate of pure butter-fat is collected and poured into a specific gravity bottle. The specific gravity bottle is a small bottle of thin glass, fitted with a glass stopper perforated by a thermometer which registers the temperature of the contained liquid, so that this may be known at the moment of weighing. This bottle must be accurately filled and then stoppered, care being taken that no air-bubble, or empty space, is allowed to remain between the stopper and the liquid, or the results will be untrue.

4. The temperature at which the fat is poured into the specific gravity bottle is a little above 100 deg. F., and then the bottle and its contents are transferred to the balance and weighed.



FIG. 57.—A specific gravity flask.

But it must be borne in mind that the precise weight must be taken when the thermometer registers exactly 100 deg. F., and the flask is entirely filled with the fat, and there is no evidence of air bubbles—a matter generally of some trouble.

The weight of the specific gravity bottle when completely filled with distilled water and closely stoppered, at the tem-

perature of 100 deg. F., has been previously estimated, and is known. By a comparison of the respective weights of the two fluids in the flask at the same temperature, the specific gravity of the butter-fat is estimated—that of distilled water being taken as 1000,

$$\text{i.e.,} = \frac{\text{The weight of the fat at 100 deg. F.}}{\text{The weight of the water at 100 deg. F.}} \times 1000.$$

#### NOTES UPON THE PROCESS.

100 deg. F. is selected as the temperature for weighing because it is the lowest temperature to which it is quite safe to reduce the contents of the bottle without any solidification of its contents ensuing—all the fats, animal and vegetable, used as adulterants, remaining liquid at that temperature.

Supposing the specific gravity is found to be 907, then if we take the lowest specific gravity which pure butter may possess as 910 the percentage amount of foreign fat in the sample may be roughly gauged.

910 = The lowest specific gravity of pure butter-fat.

904 = The highest specific gravity of pure foreign fat.

A difference, therefore, of 6 would certainly constitute 100 per cent. of adulteration.

The sample has a specific gravity of 907, and therefore a difference of 3, which will represent (6 : 3 :: 100 :  $x$ ) 50 per cent. of adulteration with foreign fat.

The specific gravity of butter-fat at 100 deg. F. is never below 909.8, and rarely below 912; that of beef and mutton fats is never above 904; that of cotton-seed oil is above 912.

**Køttstorfer's process** affords a useful means of helping one to decide as to whether the sample is true butter-fat or not. The process consists in making a direct titration of the amount of potash required to effect saponification, and it can be completed in half an hour. About 2 grammes of the clear

fat obtained in the manner already seen in performing the specific gravity test are placed in a small flask, and 25 c.c. of an approximately seminormal solution of caustic potash in highly rectified spirit are added. The whole is then gently heated in a water-bath, and stirred with a glass rod, until the fat is completely dissolved; the mouth of the flask is fitted with a cork which transmits a vertical glass tube 3—4 feet in height, and which acts as a reflux condenser, and for 15 minutes such heat is applied as will make the alcohol boil gently. The contents of the beaker are again well stirred with the glass rod (which is washed in alcohol prior to its removal) and then diluted with warm water. The strength of the potash solution having been controlled by the seminormal HCl, it now only remains to calculate from the amount of seminormal hydrochloric acid (18.185 grammes per litre) required to neutralise the alcoholic potash remaining in the flask, how much of the latter has been required for complete saponification of the butter-fat, *i.e.*, from the difference between the amounts of hydrochloric acid required by 25 c.c. of standard alkali and the amount used in the above titration the amount of potash combined with the acids of the fat is calculated. The indicator employed by Koettstorfer was 1 c.c. of an alcoholic solution of phenol-phthalein, which parts with its crimson colour when the alkaline solution becomes a neutral one.

*Example.*—1.622 grammes of clear fat required 0.36779 gramme KHO for saponification.  $\therefore$  1 gramme requires 226.8 milligrammes of the KHO for saponification.

A gramme of butter-fat requiring from 225—232.5 milligrammes of KHO for saponification may certainly be passed as genuine by this process, and it is extremely rare that fresh genuine samples of butter-fat have been found to require less than 221.5, although as low as 213.4 has been estimated. None of the other fats used as adulterants require as much as 200 milligrammes; oleo-margarine (beef and mutton fat)

generally requires about 195—197; cotton-seed oil, 191—196; nut oil, 190—196; and sesame oil, 189—190.

It is necessary to make frequent controls of the potash solution as it rapidly changes in strength.

Considerable admixture (20 per cent.) of certain foreign fats to some butter samples would not be detected by this process, and the addition of sodium carbonate to a genuine sample might lead to its condemnation.

The evidence of the presence of foreign fat which is derivable from the soluble and volatile fatty acids is undoubtedly best gained by the **Reichert-Wollny** process, and this is therefore given to the exclusion of many others.

The rationale of the process lies in the fact that the volatile and soluble fats, as has been pointed out, are relatively much higher in butter than in the other animal fats, and that when these are made to yield their corresponding fatty acids, the butyric acid formed from the butyrin is also relatively much more volatile than any of those acids yielded by the ordinary substitutes for butter-fat. If, then, this volatile fatty acid be separated, the acidity which it furnishes in the medium containing it will be considerably greater than any acidity which could be present when other animal fats have been similarly treated; and upon this fact depends the issue of the test.

1. The recently melted clear butter-fat is poured into a previously weighed small-necked round glass flask (200 c.c. capacity) until the scales register the addition of 5 grammes of butter-fat at about 100 deg. F. If fat in excess of this weight has been added, the excess may be removed by a glass rod, and with a little care it is not difficult to weigh out precisely 5 grammes.

2. 2 c.c. of a 50 per cent. soda solution, and 10 c.c. of 96 per cent. alcohol are then added to the flask, which is fitted with a vertical glass tube as in the preceding process; it is then placed in a water-bath and gently heated for a quarter of an



hour or more, the flask being gently rotated from time to time; soaps are thus formed by the combination of the fatty acids with the alkali.

The cork and tube are now removed, and the alcohol distilled off, while the flask is heated for at least half an hour. Any traces of alcohol remaining in the flask can be removed by sucking the air out through a narrow tube passed through a cork which fits the flask.

3. 100 c.c. of hot distilled water are next added, and the whole gently heated with occasional shakings until the soap is completely dissolved.

4. 40 c.c. of dilute sulphuric acid (1 in 40) are subsequently poured in after the soap solution has cooled to about 145 deg. F.; the soap is thus decomposed and the fatty acids are set free.

Re-stopper the flask, and melt the fatty acid emulsion by a gentle heat, and then let cool.

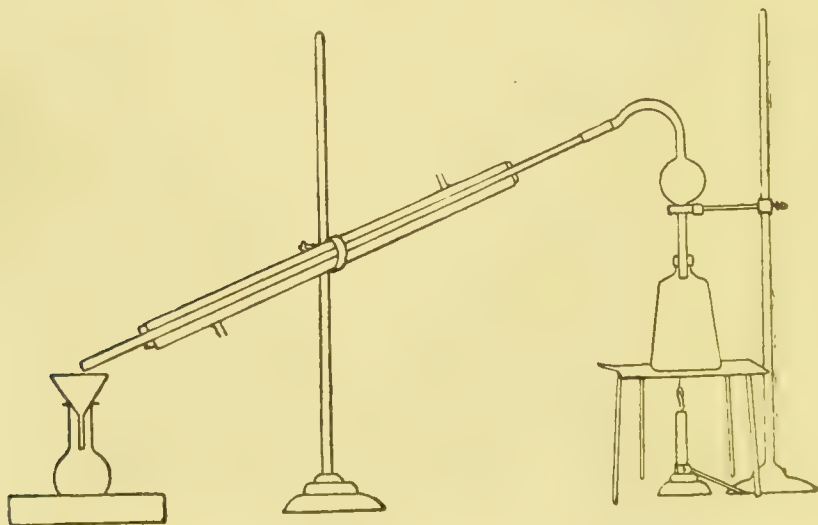


FIG. 58.—Apparatus for the Reichert-Wellny process.

5. Two pieces of pumice of the size of a pea are added to prevent bumping, and the flask is then connected to a small condensing apparatus by means of a glass tube, 7 c.m. wide,



and having at a short distance above the cork a bulb of a diameter of 2.25 c.m.; the tube is bent immediately over the bulb upwards in an oblique angle, in which direction it extends for 5 c.m., and is then again bent downward, also obliquely, and then connected with a condenser by means of an india-rubber tube. The contents of the flask are gradually heated and made to boil slowly. The insoluble fatty acids are melted and the butyric acid is distilled over unchanged; but the distillate also contains some of the insoluble volatile fatty acids, and these must be separated by allowing the distillate to run through a filter before it is finally collected.

6. Exactly 110 c.c. of the filtered distillate are collected in a graduated flask (the flame being regulated in such a way that the distillation lasts 30 minutes), and the acidity of 100 c.c. of the distillate (filtered if necessary through a dry filter paper) is estimated by finding how much of a decinormal solution of an alkali (baryta best) is required to effect neutralisation—phenolphthalein being used as the indicator.

To the figure thus obtained  $\frac{1}{10}$  is added, corresponding to the total quantity of the distillate, and the amount found by a blank experiment with the alcoholic soda solution, made at the same time and under precisely similar conditions, is subtracted. This deduction may exceed 0.2 c.c., but it should not be greater than 0.33 c.c.

5 grammes of pure butter-fat treated in this manner do not yield a less amount of acidity than corresponds to 24 c.c. of decinormal soda or baryta solution, and may require from 24—34 c.c. Oleo-margarine requires less than 3 c.c. Cotton-seed oil requires less than 1 c.c. If, then, the acidity is found to correspond to only 20 c.c. of this solution, the percentage of pure butter-fat in the sample would be as  $21 : 17 :: 100 : x$  per cent. = 80.9 per cent.; or  $100 - 80.9 = 19.1$  per cent. of foreign fat would be present.

NOTE.—By means of a T-piece in the tube by which the flask is connected with the condenser the alcohol can be dis-

tilled off and water added to the residual soap without opening the flask and exposing the contents to the  $\text{CO}_2$  of the air.

Leffmann and Beam substitute a solution of sodium hydrate in glycerol as the saponifying agent, thus shortening the time required for the process, and, it is claimed, procuring better results (*vide* their work upon "Analysis of Milk and Milk Products," pages 66-69).

**Hübl's iodine absorption test.**—This test is of use as a means of detecting the nature of certain oils and fats, and the use of vegetable oils as adulterants of butter.

**Reagents required :—**

A solution of iodine and mercuric chloride ("the iodine solution"), *i.e.*, 25 grammes of iodine in 500 c.c. of 95 per cent. alcohol, free from fusel oil, and 30 grammes of mercuric chloride in another 500 c.c. of the alcohol; filter the second solution if necessary, and then mix with the first; let stand 12 hours; always standardise immediately before use with sodium thiosulphate.

A solution of sodium thiosulphate (about 24 grammes per litre). This is standardised by means of pure sublimed iodine.

Pure chloroform—which is seen to consume no iodine on testing.

A 10 per cent. aqueous solution of iodide of potassium.

A recently prepared 1 per cent. starch paste.

Of drying oils, 0.2—0.3 gramme; of non-drying oils, 0.3—0.4 gramme; and of solid fat 0.8—1.0 is weighed out and dissolved in 10 c.c. of the chloroform, and 20 c.c. of "iodine solution" are added—the amount of iodine being such that after 2 hours the solution possesses a marked brown tint, or 5—10 c.c. more of the "iodine solution" must be added.

Having digested for 2 hours at the ordinary temperature (55 to 75 deg. F.), run in 10—15 c.c. of the potassium iodide solution; dilute with 150 c.c. of water; titrate the

free iodine with the thiosulphate, the starch being added when the yellow coloration becomes very faint.

The amount of iodine absorbed is calculated into units per cent. of the fat, and this is termed "the iodine degree," or "the iodine number." For genuine samples of butter the iodine degree is generally about 31, but it may vary from 23.5 to 39 or 40; in no other animal fats does it fall below 50 (oleo-margarine, 50.9—54.9), and in the vegetable oils most used for adulterants it does not fall below 95. In cotton-seed oil it is 105—115, and in sesame oil, 106—108.

*The melting point.*—It is recommended that, in applying this test, the butter-fat be suddenly cooled by floating it upon iced water in a platinum dish; by this means uniform results are obtained. A little of the fat is then taken up around a clean platinum wire, which is then placed alongside a thermometer in a beaker of cold water, so that the fat is very near the bulb of the instrument. This beaker is placed inside another larger one, also containing water, and thus a water-bath is improvised—by means of which the temperature of the water in the smaller beaker can be slowly and uniformly raised by the application of heat to the water in the larger beaker. The temperature recorded by the thermometer at the exact stage at which the fat melts, is that of the "melting point."

The melting points of the different fats vary within narrow limits, owing to the variable quantities of stearin, olein, and volatile fatty acids; and this test, therefore, can never prove absolute purity, since small amounts of some of the other fats—and even large amounts of beef-dripping—would not be detected.

*The water and salt in butter* may be estimated thus:—

Weigh out 5 grammes of butter into a weighed platinum dish, well mix with 30—50 grammes of sand, previously ignited and weighed, and place for about 2 hours in a drying oven at 220 deg. F.—stirring up occasionally with a glass rod to facilitate the escape of the moisture; then reweigh,

and the loss represents water. Or 2.5 grammes of butter may be placed in a tared flat-bottomed dish, and put into the hot-air oven at a temperature not exceeding 225 deg. F., until no more globules of water can be seen on looking at the beaker from below, and until a constant weight is obtained. The loss in weight represents the moisture originally present in the butter. To estimate the salt take the water-free butter and extract fat with warm ether, after the manner described in "Milk Analysis." Dry the residue, dissolve out the salt with warm distilled water, and estimate the amount of sodium chloride from a quantitative estimation of chlorine—performed as in water analysis.

An instrument invented by M. Soudén, and termed the "*liquoscope*," provides a rapid means of detecting adulteration in butter, oils, glycerine, etc. It consists of a glass cylinder containing glycerine, into which two hollow prisms are immersed. When substances with *identical* refractive indices are placed in the prisms, and a black horizontal line upon a white background is looked at through these, the line appears continuous; but when the substances are of *different* refractive indices, the line, when thus viewed, appears broken in two—one part being at a higher level than the other.

#### MICROSCOPIC EXAMINATION.

Pure butter, under the microscope, is seen to consist of a collection of small round highly refractile oil globules, to-

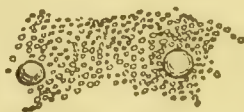


FIG. 59.—Butter-fat. ( $\times 350$ ).

gether with a few much larger ones of fairly uniform size. The larger globules average about 6 in number in the field

( $\times 350$ ) of the microscope at the same time, while the smaller would number many thousands.

In *oleo-margarine* the small oil globules are much less distinct in contour, *i.e.*, they stand out less markedly than in

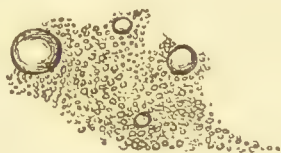


FIG. 60.—Oleo-margarine. ( $\times 350$ ).

butter-fat and have the appearance of being more crowded together; on the other hand, there is, relatively, a much greater number of the larger globules, some of which reach a much greater size than others, and these do not, in consequence, present the same uniformity as was noted in the case of pure butter.

#### ADULTERATION.

Lard, beef and mutton fats, together with vegetable oils (cotton-seed, sesame, earth-nut), are employed as substitutes of butter-fat; palm-nut and cocoa-nut oils have also been occasionally employed.

Admixture with *small* quantities of these fats is practically unrecognisable by the tests already given. The following direct tests are of assistance:—

*Cotton-seed oil*.—Mix the oil with an equal volume of a saturated solution of plumbic acetate, add ammonia, and stir quickly; the surface turns an orange red upon standing, and readily detects 10 per cent. It will be noted that this oil has a very high degree of iodine absorption and specific gravity. It has been employed as an adulterant of lard. The silver test is a good one:—1 gramme of silver nitrate is



dissolved in 100 c.c. of 95 per cent. alcohol, and then 20 c.c. of ether and 1 drop of nitric acid are added; if 2 c.c. of this reagent are mixed with 10 c.c. of the oil, and the test-tube then stood in boiling water for a few minutes, the contents blacken in the presence of cotton-seed oil.

*Cocoa-nut oil.*—The best means of detecting this oil is to take advantage of the fact that it yields considerable soluble volatile acids, which have a peculiar odour; the specific gravity will also be above that of pure butter-fat. It has not been much used on account of its strong odour.

*Earth-nut oil.*—As little as 1 per cent. furnishes a dark brownish-red colour with concentrated sulphuric acid, whereas pure butter gives a straw yellow to a reddish-yellow colour.

*Sesame oil.*—Dissolve 0.1 gramme of cane-sugar in 10 c.c. of strong HCl, then add 20 c.c. of the oil; shake and let stand; a crimson colour appears if sesame oil is present. The oil has both a high iodine absorption and a high specific gravity.

*Margarine* may be suspected if on burning a small portion of the suspected substance on a clean platinum spatula the peculiar odour of burnt tallow is given off after extinguishing the flame. Again, if 3 c.c. of the melted fat is mixed with 3 c.c. of the glacial acetic acid (S.G. 1056.2) in a test-tube, and a thermometer inserted, margarine does not form a clear solution when the mixture is warmed and continuously shaken up, until 201 deg. F. is reached, but butter clears at about 91.5 deg. F. (Valenta test). Genuine butter samples vary as to the temperature at which they clear, and the variation falls between 86—102 deg. F., but no animal fats clear below 201 deg., and no vegetable oil of common use clears below 176 deg. The test may be applied by discontinuing the heat after complete solution has taken place, retaining the thermometer in the solution, and taking the temperature at which the liquid becomes turbid. When butter is heated in a platinum



dish over a gas burner it foams considerably, and may run over the dish; but there is no noisy sputtering, as is commonly the case with margarine, and there is very little foaming with the latter.

Water is sometimes worked into a butter in excessive quantities for fraudulent purposes, but water exceeding about 16 per cent. is decidedly prejudicial to the keeping powers of the butter; such a practice increases the weight, and is most often resorted to in those cases where a ready market is ensured, and the keeping powers of the butter are not, therefore, taxed before sale. Common salt is added to improve the flavour, and also to preserve the butter by checking the decomposition of the casein; the point is not one of public health import, and rarely does the percentage amount exceed that which will lend a palatable amount of saltiness to the butter, *i.e.*, about 5 or 6 per cent.

Annatto, and more rarely turmeric, saffron, marigold, carrot, and certain coal-tar colours are used; their presence may be detected by shaking up about 5 grammes of the melted and filtered butter in a tube with 25 c.c. of a mixture of 15 parts of methylic alcohol and 2 of carbon bisulphide; the fat dissolves in the carbon bisulphide, and the alcohol, along with the colouring matter, floats above. To ascertain the nature of the agent employed it should be dissolved out by alcohol, etc., and special tests applied, as indicated in Part V., Chapter XII.

C. F. Cassal first pointed out that boric acid was frequently to be found in butter and cream, and that it plays an important part of the composition of the numerous proprietary nostrums sold for preserving these articles. Salicylic acid has also been employed (for tests *vide* Milk Analysis).

## CHEESE.

Cheese consists of the original constituents (chiefly the casein and fat) of the milk from which it is made ; but, as the process of ripening proceeds, the sugar becomes changed (chiefly into lactic acid), and the decomposition is accompanied by a diminution of fat globules and a considerable growth of bacteria, fungi, moulds, etc.

The average composition of Stilton,\* Gorgonzola, and Dutch cheese—as shown by Bell's analysis, is:—

	Water.	Fat.	Casein or nitro- genous matter.	Free acid as lactic acid.	Ash.
Stilton . . .	23'57	39'13	32'55	1'24	3'51
Gorgonzola .	31'85	34'34	27'88	1'35	4'58
Dutch . . .	41'30	22'78†	28'25	0'57	7'10

There is remarkably little *adulteration* practised in the manufacture of cheese, and the cause is not far to seek, for the article does not readily lend itself to sophistication. For the curd, which is separated from milk by rennet, there is no spurious and cheap substitute which can be made to yield the peculiar characters of pure cheese ; lard, and animal and vegetable fats have, however, been employed in the manufacture of the cheaper cheeses. The substance known as “filled cheese,” which has recently been placed upon the

\* Different samples of each of these cheeses vary considerably in the percentage amounts of their constituents.

† Very poor because made from milk which has been partially skimmed.

market, is prepared from skimmed milk, lard, and other (foreign) fats.

In this country, at the present day, it may be said that with the exception of colouring matter—which is generally annatto and harmless—the cheeses produced are almost invariably pure. It has been shown that in some cases (especially foreign cheeses) their surfaces have been brushed over with highly poisonous antiseptic solutions—such as arsenious acid and sulphate of copper—in order to preserve them from attack by parasites, and from decay; that colouring matters have also been used to tint the rind; and that those small and delicate cheeses which are wrapped in thin lead papers may take up the metal. A careful examination, therefore, of the rind for metallic poisons may be occasionally desirable.

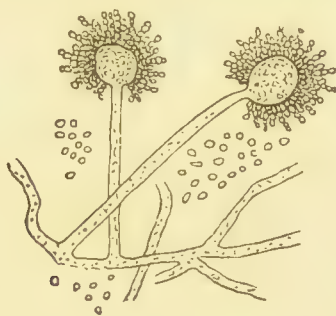


FIG. 61.—*Aspergillus glaucus* ( $\times$  about 200).

For the purposes of analysis about 10 grammes should be thoroughly pulverised in a small mortar, weighed out, and the water, fat, and ash estimated on lines already indicated. The Reichert and other tests should be applied to the fat in order to see whether it is true butter-fat.

Cheese is peculiarly liable—and especially the moister kinds—to *parasitic growths*. They are probably harmless when ingested with the cheese; but a knowledge of the commoner forms of growth is desirable:—

*Aspergillus glaucus* is a form of vegetable fungus, and

gives rise to the appearance popularly known as "blue mould"—and sometimes also of "green mould"; under the microscope its appearance is that denoted in fig. 61.

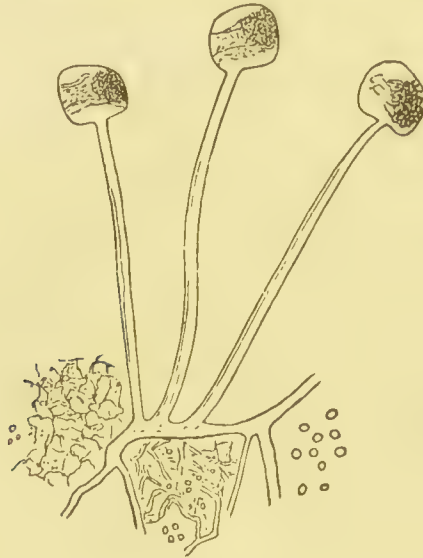


FIG. 62.—*Mucor Mucedo* ( $\times$  about 200).

*Sporendonema casei* is a similar growth, creating the appearance known as "red mould." *Mucor mucedo* is another fungus which also attacks cheese.

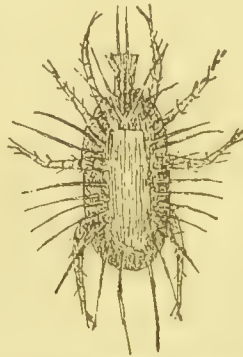


FIG. 63.—The cheese mite (*Acarus Domesticus*) ( $\times$  about 40).

*Acarus Domesticus*, the cheese mite, is a tiny animal parasite shown magnified in fig. 63.

*The cheese maggot* is an animal parasite of much larger growth; they are the larvæ of a fly known as "*piophilæ casei*," and are readily detected by either the naked eye or by the use of a small hand lens.

### TYROTOXICON.

Milk, cream, butter, cheese and ice-creams, etc., may all rarely contain this poisonous substance, which develops under those circumstances most conducive to fermentative changes generally:—viz., warmth, impure and confined air, and deficient light. It is a diazo-benzene-butyrate, and is found to occur under conditions of improper storage in the various food articles above-mentioned. It will not be necessary to examine these as a *routine* practice for tyrotoxin; but when the ingestion of ether has given rise to gastrointestinal disturbances, then a search should be instituted for this most powerful poison. The symptoms it creates commonly pass off within a few hours, but occasionally serious consequences have arisen—such as the development of symptoms akin to atropine poisoning—which may be followed by fatal collapse. The general appearance of the milk, etc., is not necessarily altered in any way, but acidity is always marked, and where this is normally present, as in cheese, it is invariably increased.

The *method of examination*, if milk be taken, is as follows:—

1. The filtrate from the milk is first rendered distinctly alkaline by means of sodium carbonate; then an equal bulk of pure ether is added and the whole well shaken up in a separator.

2. The mixture is next allowed to stand until all the ether is found to have separated into a layer upon the surface.

3. This ethereal layer is then decanted on to a saucer, where



it is left until the ether has spontaneously evaporated and a comparatively dry residue remains.

4. The residue is carefully dissolved in a little pure water, and then filtered to free it from fat.

5. The filtrate is next well shaken with an equal bulk of pure ether, and the ethereal layer, having separated, is removed and allowed to again evaporate spontaneously.

6. The residue left upon the saucer will then contain any tyrotoxicon, which may have been originally present, sufficiently pure to respond to tests—the best of which is:—

7. Pour on to the residue a few drops of a mixture of equal parts of pure carbolic and sulphuric acids—when, if the poison be present, a reddish colour appears.

In the case of cheese and butter, these are first thoroughly worked up (trituated) with water, and the filtered extract is then treated in the manner indicated above.

**Lard** is the fat obtained from the interior of the abdomen of swine; it is considerably adulterated with the oils and fats that are employed as butter substitutes (*quod vide*).

## CHAPTER V.

## CORN—WHEAT-FLOUR.

## CORN.

THIS term includes the seeds of cereal plants in general, and it is of importance to be able to distinguish between these when in a whole or ground state.

The appearance of the complete seeds is so generally well known that a description is not required here, and it is, moreover, almost entirely in the form of "flour"—*i.e.*, when these



FIG. 64.—The corn weevil (*Calandra granaria*). ( $\times$  about 40.)

seeds are finely ground—that the Health Officer or Analyst is concerned with them. Before, however, dismissing the subject, certain abnormal conditions of the entire seed which are brought about by small **animal and vegetable parasites** should be considered.

Those seeds presenting minute round perforations, and consisting almost entirely of a shell, show that the seed has been penetrated and its bulk removed, generally by a small insect, visible to the naked eye, termed *calandra granaria* (*vide* fig. 64), and popularly known as the "weevil."

Those which are small, black, and discoloured, and in which the bulk of their substance is replaced by a fine cottony material, have been attacked by the "ear-cockle," or *vibrio*

*tritici*—a small worm-like parasite, pointed at either end as shown in fig. 65; they are often to be seen in considerable numbers in damp wheat.



FIG. 65.—*Vibriones tritici*. ( $\times$  about 40).

The *acarus farinæ* is a small microscopic parasite which infests the grain, and closely resembles the *acarus scabiei*.



FIG. 66.—A wheat spikelet with ear-cockle. ( $\times$  5).

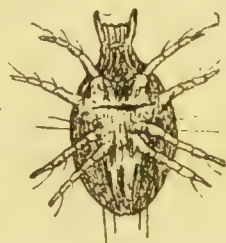


FIG. 67.—The wheat mite (*Acarus farinæ*). ( $\times$  85.)

It especially affects damp and inferior flour. Its characters are shown in fig. 67. The eggs of the parasite are oval.

The various *fungi* which attack corn are the following:—

1. *Oidium abortifaciens*, or “ergot,” most frequently attacks rye (“ergot of rye”), and causes when prevalent in the corn, a condition known as “ergotism” among many of those who habitually consume rye bread and biscuits. Those seeds which are not absolutely replaced by the fungus are discoloured brown, as is also the flour—which generally has a peculiar sour odour. A microscopic examination shows.

a very dense tissue formed by dark polygonal cells filled with oily constituents, and to the naked eye the ovary is black externally and spongy internally.



FIG. 68.—A. Ear of Rye with ergot—the latter shown as germinating and producing *Claviceps purpurea*; B. A slice of ergot. ( $\times$  about 250).

Chemically, its presence in flour may be detected by one of three methods:—

- (a) The flour is made into a paste with a weak solution of potassic hydrate, and then dilute nitric acid is added to slight excess. When the whole is subsequently neutralised by a little more of the potassic hydrate solution, a violet-red colour forms if ergot is present, and a violet colour is established when more of the alkaline solution is added.
- (b) On the addition of potassic hydrate solution to the flour, a distinct herring-like odour is appreciable—due to trimethylamine.
- (c) The flour is made thoroughly moist with ether, a few drops of dilute sulphuric acid are added, and the whole is then well agitated; on the addition of a few drops of a saturated solution of sodium bicarbonate a violet colour appears (Hoffman).

2. *Uredo segetum*, “smut,” especially affects barley, rye, and oats. The fine dark dust, which sometimes gives the ear of wheat the appearance of having been placed up the chimney, is inodorous, and has suggested the popular name “dust

brand" to the condition. Bread made with flour thus affected is bluish. *Tilletia caries* (*Uredo foetida*) and *Tilletia lævis* are of the same family (*Ustilagineæ*).



FIG. 69.—Smut spores (*Uredo segetum*). ( $\times 200$ .)

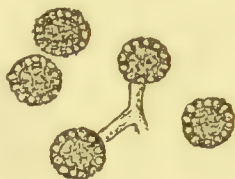


FIG. 70.—Bunt (*Uredo foetida*). ( $\times 200$ .)

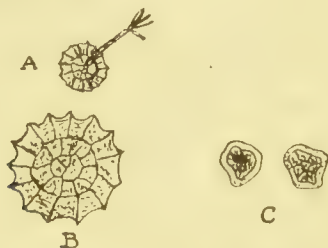


FIG. 71.—A. *Tilletia caries* (Bunt) showing germination; B. Bunt very highly magnified; C. *Tilletia lævis*. ( $\times 200$ .)

3. *Uredo foetida* (*Tilletia caries*), "bunt," affects the interior of the seeds of wheat, which it replaces by spores furnishing a fine dust, and hence the condition is sometimes called "pepper brand." The dust when rubbed between the fingers has a slippery and greasy feel, and gives off a peculiar foetid smell. No ill effect has been ascribed to the consumption of flour affected with either *Uredo foetida* or *Uredo segetum*. The microscopic appearance of "bunt" is shown in figs. 70 and 71.

4. *Puccinia graminis*.—The sporangia—as shown in fig. 72—consists of dark rounded masses, which either show a double linear contour or one presenting numerous small projections. The wheat ear and stalk are, when attacked by this fungus, more or less covered by a fine deposit, which has been most aptly designated "rust."



*Vibriones* appear in flour which is undergoing fermentative changes.



FIG. 72. *Puccinia Graminis*. ( $\times$  about 200.)

*Mucor*, *aspergillus*, and *penicillium* may also be seen in decomposing corn.

#### THE CONSTITUENTS OF THE COMMON CEREALS.

	Wheat, (winter- sown).	Barley.	Oats.	Maize.	Rye.	Rice.
Starch* . . . . .	63'71	63'51	49'78	64'66	61'87	77'66
Nitrogenous matter ( <i>i.e.</i> , albumin, cerealins, &c.) }	15'53	11'46	14'67	14'27	14'87	9'34
Cellulose . . . . .	3'03	7'28	13'53	1'86	3'23	Traces
Sugar† . . . . .	2'57	1'34	2'36	1'94	4'30	0'38
Fat . . . . .	1'48	1'03	5'14	3'58	1'43	0'19
Mineral Matter . . . . .	1'60	2'32	2'66	1'35	1'85	0'28
Moisture . . . . .	12'08	13'06	11'86	12'34	12'45	12'15
Total . . . . .	100'00	100'00	100'00	100'00	100'00	100'00

\* The starch includes from 1 to 1'5 per cent. of dextrin, and, together with cellulose and sugar, comprises the carbo-hydrates of the cereals.

† The saccharine body is allied to cane sugar in its reactions.

The numbers in the above table have been taken from Bell's analyses, and it may be pointed out that the results of the analyses of different recognised authorities all differ somewhat.

It will be seen that barley is—comparatively to wheat—poor in nitrogenous matter and sugar, but rich in cellulose and mineral matter; that oats are exceptionally rich in cellulose and fat, possess a high amount of mineral matter, but are relatively poor in starch; that maize possesses a high amount of fat, but the cellulose is low; that rye is exceptionally rich in sugar, and in other respects closely approximates to wheat; and that rice is rich in starch, but poor in everything else.

#### WHEAT-FLOUR.

The *average* composition of wheat-flour appears, from a large number of samples analysed, to be the following:—

Starch, dextrin, and cellulose	...	73
Nitrogenous matter*	... ..	10.5
Fat	... ..	1.5
Mineral matter	... ..	0.8
Sugar—corresponding to cane-sugar		0.7
Water	... ..	13.5
		<hr/>
		100.0
		<hr/>

#### THE ANALYSIS.

*Physical characters of flour.*—The colour should be white, and the flour clean; a yellow hue denotes age or fermentation, and fermenting flour disarranges the digestive system, producing flatulence, dyspepsia, diarrhœa, etc.; there

\* The nitrogenous matter varies from 8 to 15 per cent., of which gluten forms from 8 to 12 per cent.

should be no acid or mouldy smell, and practically no taste—certainly none of acidity or mustiness; taken up in the fingers the flour should be smooth, soft and coherent, with no lumpy or gritty feel, and a little flecked on to the wall should mostly adhere; on mixing with a little water, the dough should draw out into stringy masses and knit well together.

There must be complete freedom from fungi and other parasitic growths. If flour is stored in a damp place the number of microbes increases rapidly, and poisonous alkaloidal products may result from prolonged storage under such conditions. There is one great peculiarity in the chemical composition of wheat-flour, a peculiarity which arises from the large amount of *crude gluten* it contains; and it is to this substance—or rather to one of its constituents termed “gliadin”—that the characteristic adhesiveness of the flour, which makes it so peculiarly adapted for bread-making, is due.

If flour is made into a dough with water and then the dough be thoroughly washed, it is this crude gluten which remains as a sticky mass behind; the starch and other soluble substances (*i.e.*, sugar, soluble albumin, and salts) being washed away. It is of great value, therefore, both as a test of the purity and also of the quality of the flour, to estimate the amount of this substance; the best way to effect this is the following:—

Weigh out a quantity of flour—say 50 grammes—place it in a small basin, and carefully mix it with luke warm water by means of a glass rod into the condition of dough; then slowly and thoroughly work up the dough with the fingers, either under water or while allowing a gentle stream of water to fall upon it. As the dough becomes more and more washed, the water, which is being constantly emptied away and renewed, gets clearer and clearer, and the dough more stringy and sticky. A fine muslin bag may be conveniently

used in washing the flour. Ultimately the starch and all the soluble materials in the original flour are carried away, and the water escapes in a perfectly clear condition. Nothing then but crude gluten, containing generally a fraction over one per cent. of fat and salts, remains, and the entire absence of starch can be proved by treating with a little iodine. This should then be spread out in a tared (weighed) dish (or a crucible lid is convenient), the gluten dried in the water-oven, and finally weighed.

If the gluten is less than 8 per cent. the flour is not pure wheat-flour, and if it cannot be drawn out into long fine threads without breaking it is poor in quality. Rye yields a plastic gluten which cannot be separated by washing.

The *water* of flour should not exceed 18 per cent. by weight, since more than this, besides throwing up the weight fraudulently, impairs its keeping power—favouring as it does the development of fungi and the acetic and lactic acid fermentations, both of which may produce gastro-intestinal disturbances. The amount of moisture is, of course, ascertained by drying a weighed quantity of flour over the water-bath (and subsequently in the hot air oven) and noting the loss as due to water.

The *ash* of wheat consists chiefly of phosphates of potassium, magnesium, and calcium, but also of salts of silica, sodium, and iron, etc.; the total amount should not much exceed 1 per cent.; as much as 2 per cent. would imply that mineral adulterants have been added. In making the estimation, it is simply a matter of cautiously incinerating a weighed quantity of dried flour in a platinum dish, until a clean white ash remains. A short analysis of this ash can then be made if desirable.

#### **Adulteration.**—

The *foreign* mineral matter of flour may be readily and roughly estimated by shaking up with chloroform—when the

flour floats, and most of the added mineral matter settles at the bottom of the vessel. The process is repeated, in order that it shall be as inclusive as possible, and the sediment is then collected and dried, after which it may be weighed and analysed.

The presence of added minerals (calcium sulphate and carbonate, etc) is readily detected in the ash, for this is of necessity found to be exceptionally high; an analysis will disclose the nature of the adulterant.

The question of the addition of alum is dealt with under Bread (Chapter VI.), since this addition to the flour is most generally made in the process of bread-making; the test there given, however, is of equal applicability to flour.

It is chiefly by the addition of other flours and meals that sophistication is practised, and of course the cheaper varieties are selected, viz., pea, bean, barley and rice.

Old flour is occasionally passed through the mill with fresh flour; in these cases there is marked acidity, and a diminution in the fat and in the quality of the gluten.

The seeds of the Darnel grass, or *Lolium temulentum*, are apt to gain access to wheat or oat-flour; they are said to possess narcotic poisoning properties; unfortunately neither the starch grains nor the testa are characteristic—since both resemble oats very closely. The addition of alcohol, however, causes a greenish colour to appear, together with a peculiar repulsive taste, if flour contains these seeds.

The corn-cockle (*Agrostemma githago*) consists of large dull black seeds, showing small protuberances. They are markedly poisonous.

## CHAPTER VI.

## BREAD.

BREAD is made chiefly from wheat-flour, but also from rye, barley, oats, maize and rice; it is to wheaten bread that the following chapter refers.

In the process of cooking some of the starch of the wheat-flour is converted into maltose.

The composition of good bread (freed from moisture) is roughly as follows:—

Starch, dextrin, etc.	...	...	80
Nitrogenous matter	...	...	12
Maltose ..	...	...	5.8
Fat	...	...	0.5
Salts	...	...	1.7
<hr/>			
			100.00
<hr/>			

The crumb of new bread contains about 42 per cent. of moisture, and the crust about half this amount.

There are various means now employed for obtaining the porosity of bread; these are harmless and do not concern us here, with, perhaps, the exception of the very general use of **baking powders**—which consist most generally of a mixture of sodium bicarbonate, tartaric acid, and rice flour.

The tartaric acid baking powders are by far the most common, but powders are also sold in which the acid constituent is furnished by phosphoric acid, and in other cases by the sulphuric acid contained in some form of alum salt. It has



been held that the employment of acid phosphates is of value as replacing the phosphates lost to the bread by the removal of the bran. It has been ruled that baking powder is not an article of "food," under the Sale of Food and Drugs Act, 1875.

The use of baking powders containing alum should be condemned; the reaction between potash, alum, and sodium bicarbonate has been shown to result in the production of aluminium hydrate, sodium sulphate, potassium sulphate, carbonic acid and water; the hydrate of alumina appears to be dissolved by the gastro-intestinal juices.

A baking powder recently analysed by the writer gave 23 per cent. sodium bicarbonate, 33 per cent. alum, and 44 per cent. ground rice, etc. (to keep the powder dry, and prevent chemical action setting up until the powder was used).

#### THE ANALYSIS.

The bread should be fairly dry, light and spongy (*i.e.*, uniformly occupied by minute empty cells), and no hard cores should be present. It should be clean, and of a good colour—nearly white that is to say, for a yellow or dirty colour betrays age and poorness in quality. A peculiar violet tint is given to wheat containing *melampyrum* and other species of *scrophulariaciæ*, and *trefolium* (trefoil); other plants sometimes give the bread a dirty blue appearance (*rhinanthus*, etc.); *agrostemma* (corn-cockle) furnishes a greenish tint. *Oidium aurantiacum* has caused epidemics of poisoning in France; it is a reddish-yellow mould, giving a bitter taste and offensive odour to the bread. Bread must possess a pleasant taste, and should not be sodden, acid or musty.

The general principles of the analysis are similar to those of flour, and it is unnecessary to repeat what has been already given under that heading with regard to the estimation of

water and mineral matter. In the case of bread, 50 grammes of the crumbs is a convenient amount to work with.

*The moisture* in bread should not exceed 40 per cent.

*The ash.*—The increase in weight of the ash of bread over that of the original flour, is due to the common salt and the baking powder which are added in the process of baking in order to aid the dough rising. Any excess of ash—*i.e.*, above 3 per cent.—is due to added minerals, such as gypsum, chalk, or magnesium salts—added with the object of improving the colour. The ash is normally neutral, but alkaline if sodium carbonate, etc., have been added.

To estimate the silica, the ash should be treated with strong hydrochloric acid, then a little distilled water, and boiled; next filter through a Swedish filter paper, wash the platinum dish by boiling more distilled water in it, and filter these washings also through the same paper. The platinum dish being perfectly clean, well wash the material upon the filtering paper with small quantities of hot distilled water; dry in the water-oven, and then ignite in a porcelain crucible with lid; finally weigh the ash and deduct the weight of the filter-paper ash—the difference may be calculated as silica. It should not exceed 0.2 per cent.

**Adulteration** is of the same nature as that of flour, but is carried on to a greater extent, and more especially is this the case with regard to foreign flours—since the process of baking so alters the characters of the starch granules that detection is sometimes impossible. Alum, however, is the adulteration for which it is of most importance, hygienically, to examine the bread.

Mashed potatoes are looked upon as a legitimate addition, in slight amount, where sponginess is dependent upon fermentation, since they favour this action. In large quantity they are added, when cheap, to increase the weight; and since they contain between 70 and 80 per cent. of moisture they help to keep the bread moist.

Pea, bean, etc., starches may sometimes be detected by the microscope, and these would rank in every sense as adulterants, for they are not of equal nutritive value to wheat-flour, and they are added either on account of their comparative cheapness or in order to give weight to the loaf. Pea flour, nevertheless, is rarely added in any but small quantities, or it darkens the bread. If the flour is drenched with boiling water, the presence of pea and bean becomes perceptible to the sense of smell.

Rice when added also serves the purpose of giving a good white colour to the loaf.

Dr. Alford recorded a case of lead-poisoning, affecting from 15 to 20 persons, arising from the consumption of flour which had been ground by a mill-stone in which large spaces had been filled in with lead.

**Microscopic examination.**—The starch grains of the flour used in the manufacture of bread become so altered by the process of cooking, on account of the rupture of their



FIG. 73.—*Penicillium Glaucum*. ( $\times$  about 200.)

envelopes, as to lose most of their characteristics ; and hence, so far as these are concerned, a microscopic examination of the original flour should be made—indeed the whole analysis of flour is so much more easy and satisfactory than that of bread, that an endeavour should generally be made to secure samples of the former.

Fungi may be discovered, and notably the different forms

of penicillium ("mildew"), which will create, if in quantity, patches of greenish, brownish, or reddish discoloration; *oidium aurantiacum* creates an orange hue. These fungi should condemn a bread off-hand, for they may give rise to considerable gastro-intestinal disturbance if consumed in sufficient quantities.

Alumina exists normally in the pure flour, as the silicate of alumina; but it is also added, as **alum**, to inferior flour, in cooking, so as to check the fermentative action whereby a large amount of sugar (glucose) is formed, and a discoloured bread, unpleasant to the palate, results. Alum thus improves the taste and colour of the bread, and also to some extent its porosity; it is generally employed in quantities of about 15-35 grains to a 4lb loaf, but more than 100 grains have been separated.

At the present day, owing to improved processes of bread manufacture, alum is very little employed, and it is comparatively rare that alumina is detected in amounts which denote an excess over that which may be *normally* present. Large quantities of the salt were formally added to flour, and the great decline in its use commenced with the passing of the Food and Drugs Act, 1875.

It is pretty generally held in this country that *no* addition of alum should be countenanced, and that very small amounts in an article like bread, of which such large quantities are consumed, may be deleterious to health, and induce dyspepsia, constipation, etc. There is, however, some diversity of opinion as to whether small quantities of alum are injurious to health, and on this account most of the successful prosecutions under the Sale of Food and Drugs Act have been taken out under the sixth section of the Act, where the question of injury to health does not arise.

The best *test* for the presence of alum, and one which will detect very small traces (as little as one grain per lb).

is the following; but the bread must not have undergone acid fermentation.

**Reagents required.**—1. A strong freshly made tincture of logwood—prepared by digesting 5 grammes of freshly-cut logwood chips in 100 c.c. of strong alcohol.

2. A solution of ammonium carbonate—made by dissolving 15 grammes of ammonium carbonate in 100 c.c. of distilled water.

About 5 c.c. of each of these reagents are added to about 30 c.c. of water, and pieces of the crumb of the bread are cut from the centre of the loaf, moistened with a little water, and left to soak in this mixture for a few minutes; the fluid is then drained off and the bread gently dried.

The presence of alum is denoted by the appearance of a permanent lavender or violet colour, according to the amount present, while the parts of the bread which contain *no* alum are first stained the bright colour of the logwood solution and afterwards change to a dirty brown tint. Wynter Blyth soaks the bread paste in gelatine, and then tests this with logwood and ammonium carbonate; a neater reaction is thereby obtained. Whereas the presence of alum may be thus detected in small traces by a careful operator, yet considerable difficulty is experienced in estimating its *amount*. No colorimetric test applied to a small portion of the loaf can be considered as aught but crude and unreliable, since one generally sees the alum unevenly distributed about the bread—that is to say, the mixing with the flour has been roughly executed, with the effect that specks here and there may show a lavender tinge, whereas considerable areas elsewhere may be free. The operator must be careful, moreover, that he is not led astray by magnesium salts, which are capable of creating a lavender tinge almost identical with that of alum; the colour created by these salts is certainly not so permanent upon drying as that furnished by alum, but it is difficult in making an analysis



to appreciate the true value of this fact and turn it to strict account. For these and other reasons, therefore, it is necessary to resort to the difficult and tedious process of separating the alum, and estimating it by ordinary methods of quantitative analysis. In brief, a weighed quantity of the crumb removed from different parts of the loaf is evaporated to dryness, incinerated, and the ash treated with hydrochloric acid; precautions having been taken to separate any silica or iron which may be present, the alum is precipitated (and weighed) as phosphate of alumina in the presence of ammonium acetate and excess of acetic acid—the solution being boiled and filtered hot.

When it is considered how unevenly the alum is frequently distributed about the bread, it seems that the only way of achieving an *accurate* estimation must be by extracting the alumina from the *bulk* of the loaf. Undoubtedly one of the best methods of separating and estimating the alumina in bread is *Bell's modification of Duprè's method*, which cannot be better given than it is in the work upon *The Chemistry of Foods*, by Dr. James Bell.

“One hundred grammes of flour, or the crumb of bread, are carefully incinerated in a platinum capsule over a Bunsen burner, until the ash is nearly white. A quantity of the mixed carbonates of potassium and sodium, known to be free from alumina, in proportion equal to about four times the weight of the ash, is then added, and the whole thoroughly fused. The fused mass is dissolved in excess of hydrochloric acid, and the solution afterwards evaporated to dryness, and gently ignited to render the silica perfectly insoluble. Dilute hydrochloric acid is next added to the residue, which is then heated. The solution is afterwards filtered, and the insoluble silica left on the filter ignited and weighed. To the filtrate ammonia is added until a slight permanent precipitate is produced, which is then re-dissolved by a few



drops of strong hydrochloric acid. A slight excess of acetate of ammonia is next added, and after the appearance of the precipitate has been carefully noted, the mixture is raised to the boiling point, at which it is maintained for a few minutes, and then set aside for several hours. If the appearance of the precipitate becomes more granular during the boiling, or increases very largely in quantity, as will sometimes be the case, it is evident that phosphates of lime and magnesia have been precipitated, and the precipitate must be filtered off, dissolved in a little hydrochloric acid, warmed and reprecipitated as before with acetate of ammonia solution. The precipitate is filtered off, washed, and again dissolved in a small quantity of hydrochloric acid. The solution is boiled for a few minutes with about five grains of bisulphide of soda, and again for a few minutes longer after the addition of an excess of pure caustic soda. The precipitate of oxide of iron is filtered off, and the filtrate slightly acidified by hydrochloric acid. Acetate of ammonia in slight excess is then added, and the solution raised to the boiling point, and allowed to stand for several hours. The resulting precipitate, which should consist of pure phosphate of alumina, is washed, dried, ignited, and weighed. The weight of the precipitate multiplied by 3.873 or 3.702 gives the amount of potash-alum or ammonia-alum respectively, corresponding to the total alumina in the one hundred grammes of flour or bread taken. Before accepting the result as conclusive, care should be taken to prove that the precipitate consists of phosphate of alumina only.

“In this process for estimating alumina as originally proposed, the phosphate of alumina was precipitated in the cold, but in our experiments on flour containing known quantities of alum, it was found that the whole of the alumina of the alum was not precipitated in the cold, even after standing overnight, and that the quantity remaining in solution could

be recovered by boiling. It is true that by so doing there is increased danger of the precipitate containing phosphates of lime and magnesia, but this contingency is fully provided against by repeating the precipitation in the presence of free acetic acid and subsequent treatment with pure soda."

*Conclusions to be drawn from the amount estimated.*— If the amount of alumina represents more than from 6 to 10 grains of alum per 4lb loaf, in the vast majority of cases the latter has been fraudulently added; some pure flours, however, do undoubtedly contain a greater quantity than this as a normal constituent, and hence it is difficult to lay down any definite quantity as a standard beyond which the proof of fraudulent addition may be held to be established. The alumina which is taken up from the soil is in the form of silicate, and at first sight it would seem that, taken up naturally in this form, it could be estimated from the silica present in the ash. This point would be of great service if it held good, but analysis shows no definite and constant relation between the silica and the alumina of pure flour or bread; nor could such be expected when it is borne in mind that silicates—other than that of alumina—may be yielded up by some soils to the cereals growing upon them. If, however, the amount of alumina considerably predominates over that of the silica, there is then little doubt of the presence of "added alum." The subject, then, of "added alum" will be seen to be both an unsatisfactory and a difficult one, from the analyst's point of view; and it is satisfactory that we are becoming less and less often called upon to make the estimation.

## CHAPTER VII.

THE AVERAGE COMPOSITION OF OTHER CEREALS AND THE PULSES.\*—THE MICROSCOPIC CHARACTERS OF THE DIFFERENT STARCH GRANULES.

## OATMEAL.

Starch, dextrin and cellulose	...	63
Nitrogenous matter	...	16
Fat	...	6.25
Mineral ash	...	2
Sugar	...	1.25
Water	...	11.5
		<hr/>
		100.00

The only common *adulterant* is barley meal. The husks of other cereals are sometimes ground up with the oats, and rice and maize are said to be rarely added also.

## ARROWROOT.

Starch, dextrin and cellulose	...	83
Nitrogenous matter	...	0.8
Mineral ash	...	0.2
Water	...	16
		<hr/>
		100.0

When *adulteration* is practised it is generally by potato, sago, or tapioca starch.

\* Chiefly compiled from the results of analyses made by the writer.

## SAGO.

Starch, dextrin and cellulose	...	...	...	85.05
Nitrogenous matter	...	...	...	0.8
Mineral ash	...	...	...	0.15
Water	...	...	...	14.00
				<hr/>
				100.00
				<hr/>

The *adulterant* chiefly used is potato starch.

## TAPIOCA.

Starch, dextrin and cellulose	...	...	...	85.95
Nitrogenous matter	...	...	...	0.55
Mineral ash	...	...	...	0.1
Water	...	...	...	13.4
				<hr/>
				100.00
				<hr/>

*Adulterants*.—Potato, sago and rice are all cheaper, and are sometimes used on this account.

## CORN FLOUR\* (MAIZE).

Starch, dextrin and cellulose	...	...	...	70.5
Nitrogenous matter	...	...	...	9
Fat	...	...	...	4.5
Mineral ash	...	...	...	2
Water	...	...	...	14
				<hr/>
				100.0
				<hr/>

\* Corn flour consists of the nearly pure starch of maize or rice.

## LENTIL FLOUR.

Starch, dextrin and cellulose	...	59
Nitrogenous matter	... ..	26.5
Fat	... ..	2
Mineral ash	... ..	2.5
Water	... ..	10
<hr/>		
		100.0
<hr/>		

This and the following meals and flours are too cheap to make *adulteration* profitable.

## PEA MEAL.

Starch, dextrin and cellulose	...	58
Nitrogenous matter	... ..	25
Fat	... ..	3
Mineral ash	... ..	2.5
Water	... ..	11.5
<hr/>		
		100.0
<hr/>		

## BEAN (HARICOT) MEAL.

Starch, dextrin and cellulose	...	57.5
Nitrogenous matter	... ..	26.25
Fat	... ..	2.5
Mineral ash	... ..	2.25
Water	... ..	11.5
<hr/>		
		100.00
<hr/>		



## RYE MEAL.

Starch, dextrin and cellulose	...	71.25
Nitrogenous matter	...	11
Fat	...	2
Mineral ash	...	1.75
Water	...	14
		<hr/>
		100.00
		<hr/>

## BARLEY MEAL.

Starch, dextrin and cellulose	...	72
Nitrogenous matter	...	11.25
Fat	...	1.75
Mineral ash	...	1
Water	...	14
		<hr/>
		100.00
		<hr/>

## POTATO FLOUR.

Starch, dextrin and cellulose	...	22.85
Nitrogenous matter	...	2
Fat	...	0.15
Mineral ash	...	1
Water	...	74
		<hr/>
		100.00
		<hr/>

The potato juice has a faint acid reaction.

The potato bulb should be firm and resistant to the knife, and show no disease (fungi, etc.). The best practical test is that of cooking them, and then noting their appearance and taste.

## GROUND RICE.

Starch, dextrin and cellulose	...	82
Nitrogenous matter	... ..	5
Fat	... ..	0.5
Mineral ash	... ..	0.5
Water	... ..	12
<hr/>		
		100.0
<hr/>		

## THE MICROSCOPIC CHARACTERS OF THE DIFFERENT STARCH GRANULES.

The starch, of which the foregoing food-stuffs are mainly composed, exists in the form of microscopic granules, which are more or less characteristic of the particular plant from which they are derived, on account of their different sizes, shapes, and markings. These microscopic granules consist of an extremely thin envelope of cellulose enclosing the starch (granulose), and the latter generally appears to be arranged in fine superimposed strata—which accounts for the “striæ,” or concentric lines, commonly discernible upon the external surface of the granule.

Suppose a sample of any flour or meal is to be examined under the microscope, then a very small amount is placed upon a clean glass slide, a drop of water is applied, and a clean cover-glass is pressed firmly down over the powder and water to evenly distribute the powder. It is impossible to get *too* thin a layer of the substance in order that a satisfactory examination may be made, as otherwise granules get superimposed and conglomerated, and their contours and markings become indistinguishable. It is a good plan, therefore, to drop a small amount of the powder upon the slide,

and then to gently blow it almost all away again, before applying the water and cover-glass.

It is important that the reader should recognise that in the description which follows it is the *characteristic* cells which are described in each case. It must not be thought that in a sample of arrowroot, for instance, each granule will possess the characters described under that head; such is by no means the case; some may have the hilum in the centre, or even at the small extremity of the granule (as in potato) and yet the sample may be pure; *by far the majority* of the cells will, however, possess in a more or less marked degree, the characters described. Where, therefore, the starch grains of different food plants somewhat closely resemble each other, it is difficult to decide as to whether there may or may not be some admixture, although considerable adulteration admits of no questioning; but when these grains are dissimilar in appearance, the faintest possible amount of admixture is detected by the microscope. When it is required to estimate the *amount* of adulteration in any powdered substance, a rough percentage of the foreign starch grains may be made by counting these upon the microscopic "field" of several mounted specimens; and when the percentage amount of foreign starch present has been thus judged, a careful and thorough mixture is made up containing the supposed amounts of the ingredients in the composition under examination; this is then examined under the microscope and compared with the original powder in order to see if the estimation which has been made is broadly correct. If not, known quantities of the pure substance are mixed with fresh quantities of the adulterants found, until examination shows that the approximately true percentages have been arrived at. Or, to be more accurate, the number of foreign starch granules in the specimen may be counted upon a plan very

similar to that adopted in the case of blood corpuscles—a plan with which the reader is familiar.

It has been seen that, in many cases, the differences between the starch granules are very slight, and some skill is requisite in detecting them; such skill is only acquired from a steady use of the microscope, and the student is recommended to fit up a small case containing samples of all the more common starches, and to practise assiduously with these; in order that he may be able to better appreciate the slight differences which distinguish some of the starch granules, it is recommended that a *long* piece of glass slide is employed, so that he may mount upon it specimens of all those which closely resemble each other and compare them, almost at the same time, by simply moving the slide across the “field” of the microscope. It is useless to make any attempt at mounting these specimens *permanently*, even in glycerine, since they soon lose some of their characters. A quarter-inch power should be employed, and this suffices for all practical purposes.

Here, space only permits of mention being made of the valuable adjuncts which the polariscope and photography furnish to such investigations.

1. *Large, round or oval granules, more or less flattened, and showing no marked “striae,” or at most only at the margins; together with others extremely small and ill-defined.*

*May be wheat, barley or rye.*

*Wheat*.—Few, if any, “intermediary”\* sizes, although the larger granules themselves vary somewhat in size. (A linear hilum and striæ are visible under a high power, and the small granules are seen to be angular).

\* A term used, in this connection, to denote a size about *midway* between that of the large and small granules.

*Barley*.—Similar, but the large granules are rather more irregular in shape and somewhat smaller, and “inter-

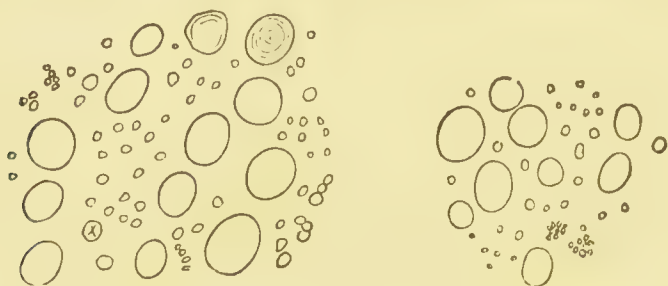


FIG. 74.—Wheat. ( $\times 200$ .)      FIG. 75.—Barley. ( $\times 200$ .)

mediary” sizes are more commonly present; lumpy forms rather more common.

*Rye*.—Similar, but many show a rayed hilum, and present cracked edges; the granules are somewhat larger, and more

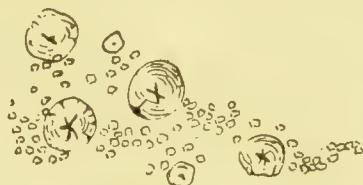


FIG. 76.—Rye. ( $\times 200$ .)

generally circular and flattened than those of wheat or barley. Rye flour is darker and less finely ground than wheat flour.

2. *Large, pyriform or oval granules, with well-marked concentric striæ, and a circular or short linear hilum.*

*May be potato or arrowroot.*

*Potato*.—Typically, they have the appearance of an oyster-valve. A well-marked circular or stellate hilum is at the smaller extremity, and the striæ are well marked. They vary considerably in size.

*Arrowroot*.—Similar, but the hilum is generally at the larger extremity, and the granules average a trifle smaller—with the exception of the arrowroot named “tous-les-mois,” in which the granules are commonly even larger than those of potato. The granules do not swell with potassic hydrate

solution as do those of potato. There are many varieties of arrowroot, all of which, however, present similar general



FIG. 77.—Potato. ( $\times 200$ .)



FIG. 78.—Arrowroot. ( $\times 200$ .)

characteristics as to their starch granules; the common variety is derived from *Maranta arundinacea*.

3. *Ovalish granules, with faint concentric striæ, and with a central linear hilum.*

*May be pea or bean.*

*Pea.*—Most have a central longitudinal hilum, which presents a puckered appearance. The granules are large.

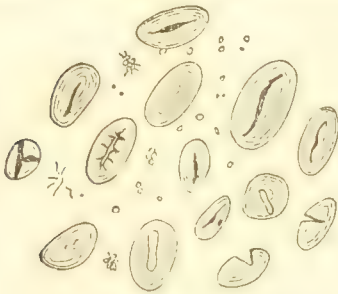


FIG. 79.—Pea. ( $\times 200$ .)



FIG. 80.—Bean. ( $\times 200$ .)

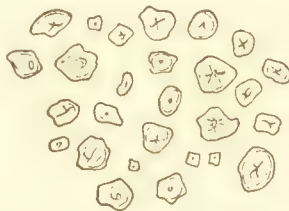


FIG. 81.—Maize. ( $\times 200$ .)

*Bean.*—Similar, but somewhat larger and more flattened.



(i.e., broader) and slightly more uniform in size. The hilum is much more commonly crossed by transverse lines "puckered").



FIG. 82.—*Bruchus pisi* (of the pea, bean, etc.). ( $\times$  about 40.)

4. *Angular and faceted\* granules.*

*May be rice, oatmeal, or maize.*

*Rice.*—The granules collect into angular masses, and are very minute.

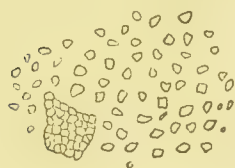


FIG. 83.—Rice. ( $\times$  200.)

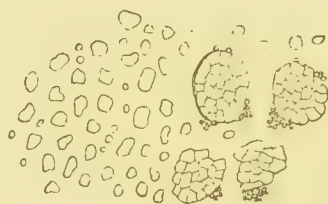


FIG. 84.—Oatmeal. ( $\times$  200.)

*Oatmeal.*—The granules collect into rounded masses, and are slightly larger than in rice, but still very minute.

*Maize.*—The granules are much larger and are more irregular in shape—which tends towards the circular; they possess a visible hilum which is generally stellate.

5. *Irregular in size, rounded or with rounded edges, possessing (generally) a central hilum, and showing ill-defined concentric striæ.*

*May be sago or tapioca.*

*Sago.*—Mostly large, and very irregular in shape; commonly elongated and round at one larger extremity, and truncated at the other. Hilum stellate or linear.

*Tapioca.*—Similar, but much smaller, and many granules

\* These facets are due to the close juxtaposition of the granules.

have a tendency to assume the flask-shape, that is, are truncated by one facet. Hilum generally more towards the rounded extremity.

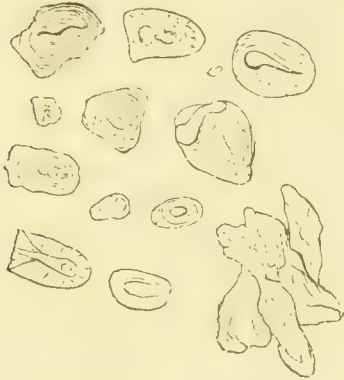


FIG. 85.—Sago. ( $\times 200$ .)



FIG. 86.—Tapioca. ( $\times 200$ .)

The so-called flours and meals derived from the cereals, in addition to the starch granules which have been described,

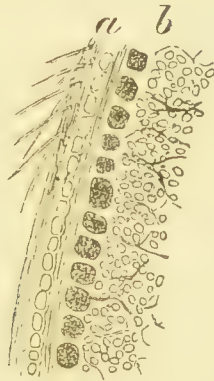


FIG. 87.—Section of wheat grain (outer coat). ( $\times 50$ .)

*a.* Girdle cells. *b.* Cereal cells.

also give evidence, here and there upon the "field" of the microscope, of the thin envelope of the grain—called the skin or testa; and this is the case with even the finest ground and purest flours.

In *wheat* the envelope is composed of three\* fine membranes, the external and the middle both consisting of flattened elongated cells, whose contours present a beaded appearance, and which are more or less dove-tailed into each other.

The long axes of the cells in the middle coat are disposed at right angles to those in the external—the latter being arranged with their long axes corresponding with that of the grain.

The external coat is made up of two or three layers of cells, the middle of only one—and the cells of the latter are smaller and more uniform in size. Unicellular cells ("hairs")



FIG. 88. — Wheat. Tissue from the "testa" of the grain. Showing the appearance of the cells forming its outer and inner membranes. ( $\times 100$ .)

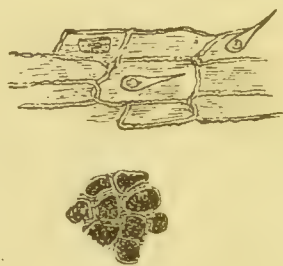


FIG. 89. — Barley. Tissue from the "testa" of the grain. Showing the appearance of the cells forming its outer and inner membranes. ( $\times 100$ .)

with pointed apices, come off in tufts from the external coat at one extremity of the grain; these "hairs" are simply prolongations of the cells.

The internal coat is made up of irregularly rounded opaque-looking cells, which frequently contain one or more oil globules. The starch granules, comprising almost the whole of the interior of the grain, are included within a thick-walled cellular net-work.

\* There are probably six in all under very high powers.

In *barley* the envelopes are the same as those in wheat, except in the following respects:—

The cells forming the external coat are smaller and more uniform in size than in wheat, and their outline is serrated instead of beaded; they carry, moreover, *short thick* hairs. The cells of the middle coat are more elongated, and not beaded—or very imperfectly so. Those of the inner coat are somewhat smaller.

Between the middle and internal coat is a layer of long narrow cells, arranged with their long axes at right angles to those forming the middle coat.

Slight as these differences are, it is to the envelopes rather than to the starch granules that one must turn in order to discriminate between wheat and barley.

In *rye* the testa so closely resembles that of wheat that it is difficult to hit upon a point in which they differ, and it is

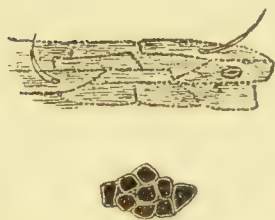


FIG. 90.—Rye. Tissue from the “testa” of the grain. Showing the appearance of the cells which form its outer and inner membranes. ( $\times 100$ .)

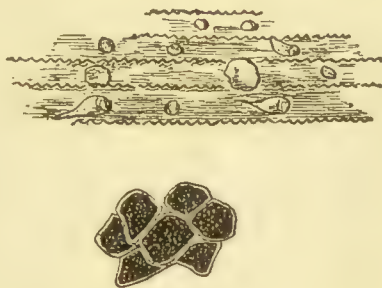


FIG. 91.—Oats. Tissue from the “testa” of the grain. Showing the appearance of the cells which form its outer and inner membranes. ( $\times 100$ .)

fortunate that the starch grains afford a ready means of distinguishing between the two.

It may be pointed out that the unicellular hairs are somewhat shorter than in wheat.

In *maize* (Indian corn) the envelopes are two in number ;

the external consists of several superimposed layers of flattened elongated cells, and the internal of a layer of cells of irregular size and shape but otherwise resembling the internal layer of wheat. What is very characteristic, however, about maize is that the cellular network, which holds the starch granules, in this plant forms an irregular mosaic—most often pentagonal but occasionally polygonal in design.

In *oats* the envelopes consist of an external one of long narrow cells with evenly serrated contours (not wavy or beaded), and carrying short sharp spinous "hairs"; a middle, somewhat similar coat, but indistinct and poorly seen; and an inner layer of cells resembling the internal one of wheat, but larger.

In *rice* the external coat of the husk, consisting of long narrow cells, is characterised by the number of fine siliceous particles it contains, and which are collected together into ridges crossing each other at right angles. The spinous hairs are long and numerous, and the other coats, of which there are several, consist also of elongated, narrow, flattened cells, variously arranged.

## CHAPTER VIII.

## MEAT. PARASITES OF FLESH.

By the provisions of sections 116, 117, 118 and 119 of the Public Health Act, 1875, Sanitary Officers are empowered to examine meat which is intended to be sold for human consumption, and if such is found to be unsound, to seize it and carry it away to be dealt with by a Justice.

When the flesh of one animal is represented as being that of another, proceedings are taken under section 6 of the Sale of Food and Drugs Act, which provides that "no person shall sell to the prejudice of the purchaser, any article of food which is not of the nature, substance, and quality demanded by such purchaser, under a penalty not exceeding £20."

When it is contemplated to seize a piece of meat, it becomes necessary for a sanitary official to make a careful examination of such, in order to decide whether it is unfit for human consumption. The question is not always an easy one to decide upon, and considerable numbers of poor and diseased animals are killed yearly and consumed, their flesh betraying few, if any, of the characters of bad meat. It is generally so much more easy to prevent the sale of diseased meat by even a cursory inspection of the animals before death than it is by any laboured investigation over a butcher's stall, or in a laboratory, that it may be hoped in the future that the slaughtering of animals will be confined to public abattoirs, where each animal as it enters is examined in a routine manner by skilled officials. It may be said in this connection, without fear of contradiction, that whereas in



almost every other respect our sanitary code in this country is considerably in advance of that of our continental neighbours, yet, in this one respect of the examination of living animals prior to slaughter and of their carcasses, we fall most lamentably behind them, and that the scant provisions at present made are a disgrace to a country which has grown—with just claims in other respects—to be regarded as *facile princeps* in sanitation.

When a piece of meat has been seized as suspicious, deep sections should be cut and examined, and in those cases where putrefaction is judged to have commenced, special care should be paid to that flesh which is more immediately adjacent to the bone.

#### THE CHARACTERS OF GOOD MEAT.

It should have a marbled appearance—due to little streaks of fat between the muscular fasciculi; the whole surface should have a glossy appearance, and the colour should be of a bright florid hue and not too dark—or the meat is that of an old or diseased animal. The colour of veal, mutton, and pork is always paler than that of beef, and this fact depends to some extent upon natural causes, but mostly upon the fact that calves, sheep, and pigs are bled at the time of killing. The flesh of all young animals is also naturally paler than that of older ones. Should the meat be very pale and yellow (“white flesh”), and the animal an adult one, fatty infiltration or degeneration, or fibroid infiltration, is probably the cause. A magenta hue points to some acute specific condition present at the time of death.

The connective tissues should glisten when exposed, and the muscular fasciculi should not be too large and coarse.

To the touch the meat should be firm and slightly elastic, which implies that the meat is fresh and has set well (rigor

mortis); it should, moreover, be so dry upon the surface that the finger is only slightly moistened by being passed over it; such moisture should be of a clear red colour, and of an acid reaction. On cutting through a muscle, the whole thickness should present a uniform colour, or the interior must be but very slightly paler than the more external flesh.

The odour of meat is best obtained either by drenching it, when finely minced, with very hot water, or by plunging a clean odourless knife deep down into its substance—preferably in the direction of bone—and then withdrawing and smelling the knife. The peculiar odour of good fresh meat is familiar to all, both in the raw and cooked state, and any departure from this would create suspicion.

The fat has a firm and greasy feel; the normal faint yellow colour must not be excessive—although the fat of animals fed upon some oil cakes acquires a very distinct yellow hue. The fat deepens in colour with age. It should present no hæmorrhagic points.

Any lymphatic glands attached should be firm, slightly moist, and of a pale greyish yellow appearance on section.

The marrow of the bones should be light red, and that from the bones of the hind quarters should set firmly within 24 hours, but that of the fore quarters remains diffuent for a longer period.

The ash of the meat is alkaline, and consists almost entirely of phosphates and chlorides.

Upon cooking, the fibres do not cook hard; the loss does not exceed 30 per cent. (whereas bad meat containing an undue amount of water may lose over 40 per cent.); and a savoury odour escapes.

Dried upon a water-bath for several hours, it does not lose more than 74 per cent. by weight.

## THE CHARACTERS OF BAD MEAT.

A deep purple or dark tint suggests that the animal has either not been killed and bled, but has died with the blood in it, and probably of some acute feverish condition, or else that death has followed upon some pulmonary complaint involving mechanical obstruction to respiration. A yellow or mahogany hue denotes bile-stained flesh.

Well-defined and dark coloured areas full of blood are due to hypostatic congestion or post-mortem staining.

Pus may be seen lying between the muscle fibres, and tumours, etc., may be present (as in anthrax, etc.).

There is frequently too great a proportion of bone to flesh, and generally the reaction of the juice (which is dark or discoloured) is alkaline or neutral. The odour may be that of putrefaction, or of a faint and sickly nature. It may be sweet (uræmia), or urinous (due to local effusions of urine).

Sometimes there is a smell of physic, as when, previous to death, odorous and volatile drugs such as camphor, prussic acid, turpentine, creosote, chloroform, etc., have been administered; or the animal may have fed upon odorous plants; or, subsequent to death, the carcase may have been hung in an atmosphere which is odorous from any cause (tobacco, carbolic acid, etc.).

It has been shown that no dangers to the meat would arise from the administration during life of medicaments such as arsenic, antimony (tartar emetic), or strychnine; but if there is any reason for suspecting the presence of any poisonous substance in the *internal organs* liable to be consumed, some of these should be given to a dog or cat, and its effects carefully watched. The animal may in some cases take in poison by its food, by feeding upon such herbs as bryony, meadow-saffron, rhus toxicodendron, etc.

Bad flesh is frequently moist, sodden, flabby and dropsical,

and infected with parasites. It must be remembered, however, that the flesh of young animals is always pale and moist.

Some parts of the meat generally feel softer than others, that is to say, there is not a uniform resistance to pressure and occasionally there may be emphysematous crackling. The flesh of veal and lamb has been blown out artificially, and the surface then smeared with fat; and thus an artificial plumpness has been given to poor meat. Dishonest butchers also sometimes rub melted fat over the flesh of diseased animals to give it a healthy and glossy appearance (Gamgee).

The fat is generally soft and flabby, or gelatinous; frequently highly coloured, and exhibiting small hæmorrhagic points.

Any attached lymphatic glands will often be either enlarged, softened, hyperæmic, ecchymosed, caseated, calcified or suppurated. The marrow of the bones is discoloured—often brownish—and sets badly.

In *commencing putrefaction* the flesh softens, and tears readily; it becomes paler; the elastic resistance gradually diminishes and becomes less uniform, *i.e.*, some parts are softer than others; the characteristic odour is developed; the marrow softens and turns brownish; and the juices become alkaline in reaction—due to ammonia, and substituted ammonias being formed by the action of schizomycetes. Later, the meat becomes of a greenish hue, and a glance then suffices to detect the presence of putrefaction. Sometimes it becomes luminous, chiefly from the presence of B. Phosphorescens.

Certain diseases cause distinct, and sometimes characteristic, appearances in the meat—and notably is this the case with anthrax and the anthracoid diseases (quarter ill, etc.), pyæmia and septicæmia; in these the tissues and organs generally are congested, the blood is dark in colour and viscid, ecchymoses and hæmorrhages are frequent and the

lymphatics are enlarged and congested ; but it is only from an examination of the internal organs that a definite diagnosis can be attempted. When, therefore, any suspicion attaches itself to the sample of meat under examination, every attempt should be made by the Health Officer to penetrate the thicket of difficulties and subterfuge which will beset his path, and to gain a glance at the offal of the animal in the slaughter-house (if not too late), and more especially to carefully inspect the liver, lungs, and lymphatics. The term "offal" includes the head, the feet, the skin, and all internal organs except the kidney ; the remainder of the animal is termed the "carcase."

*The lungs* may be the seat of hepatisation, œdema, infiltration with pus, multiple abscesses, cavities, nodules, etc., and *the pleura* may be adherent, or contain effusions—in such diseases as pleuro-pneumonia, tuberculosis, pneumo-enteritis, anthrax, glanders, swine plague, and actinomycosis ; a microscopic examination may detect specific germs.

Tuberculosis of the lungs is popularly known as the "pearl disease" or "the grapes," the pearls referring to the tubercular deposits which hang down from the surface of the lungs like grapes. The lymphatic glands of the thorax may be enlarged, caseous, calcareous or purulent.

*The liver* may be enlarged, tuberculous, softened, congested or bile-stained, and may show hydatids, multiple abscesses, nodules, or the liver-fluke, etc., in different diseases.

*The spleen* may be enlarged and with rounded edges, or contain nodules or ulcers, etc. (as in anthrax, glanders, bacillary erysipelas, etc.).

*The lymphatic glands* (abdominal) may be enlarged and inflamed, etc. (as in pig-typhoid, pneumo-enteritis, glanders, small-pox of sheep, swine plague or fever, tuberculosis, bacillary erysipelas, etc.).



*The kidneys* may be inflamed and show hæmorrhages, etc. (as in tuberculosis, swine fever, anthrax, cattle plague, etc.).

*The stomach, intestines and peritoneum* may be inflamed, congested or ulcerated (as in pneumo-enteritis of the pig, etc., swine plague, cattle plague, anthrax, etc.).

*The head.*—The tongue and mucous membrane of the mouth may show vesicles and ulcers, due to foot and mouth disease; the lower jaw, the tumours of actinomycosis; the brain may show hydatids and tuberculosis; the small cysts of trichinæ and measles may be seen in the conjunctiva, or the under surface of the tongue; the nose may be ulcerated and the membrane nodular and showing pale radiating scars, from glanders and farcy in the horse, or from small-pox in the sheep.

*The skin.*—Variola-like eruptions of spots (papules and pustules) may be present; the wool and hair rough and patchy, and showing red blotchy skin beneath (as in carbuncular inflammations, anthrax, etc.).

*The udder* in cows may show vesicles, ulcers, nodules, etc. (as in such conditions as foot and mouth disease, tuberculosis, Hendon disease, etc.).

*The feet* may show vesicles and ulcers, and the hoofs may be loose and even shed (as in foot and mouth disease).

In **frozen meat** the expressed juice shows under the microscope that the red corpuscles have dissolved in the serum, and the hæmoglobin appears as irregular yellowish-brown crystals.

**Opinion to be formed.**—It is by no means an easy matter to decide as to what shall suffice to condemn meat. Opinion at the present day is very much divided upon those diseases which render the flesh of animals attacked by them unfit for human consumption. Most authorities appear, however, to be agreed in condemning the whole of the flesh of animals



slaughtered while victims of the following diseases, either on account of the poor and altered quality of the meat, or from the risk of eating it:—

Pyæmia	}	In those cases where multiple abscesses are fairly general; or where sloughing occurs anywhere about the body; or where putrefaction has set in.
Septicæmia		
Uræmia		
Erysipelas		

Anthrax and anthracic diseases.

Cattle plague.

Advanced rabies or swine fever.

Glanders or farcy of the horse.

Hydatid diseases.

Variola of sheep.

Trichiniasis.

Chicken-cholera.

Tuberculosis and cancer, where deposit, etc., is fairly general, and emaciation is marked.

In most other diseases it is generally held to be sufficient to condemn the affected parts, if the rest of the carcase appears healthy.

The question as to whether tubercular meat can spread infection to man is still *sub judice*, but there seems little reason to doubt that it has this power; if, however, the meat is properly cooked, this danger is removed—but large joints are often insufficiently cooked throughout, and the temperature to which the deepest portions of the joint is raised may often on this account be insufficient to destroy the more resistant infective matter; raw flesh, moreover, is now largely given to infants and invalids. The organs (internal) must in every case be unhesitatingly condemned.

The subject of the spread of tuberculosis through the means of the flesh of tubercular animals has received of late—as befits its importance—considerable attention.

Tuberculosis is a very common disease in oxen, and it is

rather common in swine and poultry ; on some parts of the Continent it is found that 20 per cent. of all the cattle slaughtered are infected with this disease.

It is admitted by all that the bacillus tuberculosis may under certain circumstances be taken in by the alimentary canal ; the great frequency of mesenteric disease in young children, which is almost certainly derived in a large number of cases from tubercular milk, is an unhappy proof of the last mode of infection.

Portions of tubercles and cultures of the bacilli have, when given to various animals to swallow, produced tuberculosis.

Birch-Hirschfield and Schmoll have shown that the bacillus, in travelling from one organ to the other, must pass through the general lymphatic or blood system ; and it is impossible to guarantee that at any given time, the flesh through which these are circulating is free from infection, *although no lesions have been locally produced*. Dr. Newsholme maintains that tuberculosis is an infective disease contaminating the whole animal in every case, and there will be found few to disagree with him, for the whole of the animal's blood must be infected with the bacilli tuberculosis, and who shall say that no part of the carcase of such an animal may not be given in a perfectly raw condition to infants and invalids ?

There are many who will not eat certain kinds of meat which have not been suffered to reach advanced stages of putrefaction ; but whatever be the peculiar desires and tastes of the consumers there is no excuse for vendors offering any but *fresh meat for sale* ; in other words it should be insisted that the meat is *sold* fresh, so that whether it is cooked and eaten as such may be entirely a matter of the individual tastes of the purchasers. There is no doubt that with many, any flesh in which decomposition has only even made a start entirely disagrees with them ; and abundant evidence is not lacking, that when an advanced state of putridity has been

reached, violent gastro-intestinal irritation, followed by diarrhoea, vomiting, and toxic symptoms, are induced. These symptoms are probably due to the presence of crystallisable toxic alkaloids and toxic albumoses, which are elaborated from the albuminous material by the agency of micro-organisms ; of these substances a large number has now been extracted from putrifying organic matter, some of the most important being cadavarine, choline, collorine, muscarine, neuridine, putrescine, sepsine, and tyrotoxin. These alkaloidal products of putrefaction are separable by their relative solubility in acid, ether, chloroform, etc.

It is not desirable, in the public interest, to proceed in the matter of meat condemnation too rigorously, but rather to adopt a standard based upon careful considerations as to the actual safety of consumption ; for too extensive restrictions can but have the effect of increasing the cost of a necessary article of diet, which is already almost beyond the reach of the poorest of the community.

The subject of a microscopic examination is a lengthy one, and the pathology of the morbid tissues of cattle, etc., so closely resembles that of human beings in such common diseases as tuberculosis, pneumonia, pleurisy, etc., that nothing special in this relation need be gone into here—since the reader will be already familiar with the tissue changes, etc., which accompany each of these diseases : nor, indeed, is a microscopic examination generally called for, the macroscopic post-mortem examination of the tissues amply sufficing, in the majority of cases, for a diagnosis.

In the case of those diseases dependent upon the presence of specific micro-organisms, a careful microscopic examination is essential, and special bacteriological methods must generally be adopted. The *zymotic diseases* so-called (*i.e.*, pleuro-pneumonia, malignant catarrhal fever, erysipelas, muco-enteritis, influenza, diphtheria, croup, rabies, anthrax

and anthracic diseases, rinderpest or cattle plague, foot and mouth disease, swine fever, variola, actinomycosis, tuberculosis and glanders or farcy of the horse) are all due to the presence of specific micro-organisms.

Of those organisms which are commonly referred to as "the parasites of animal tissues," some only are inter-transmissible when the flesh is eaten by human beings, and since the sub-



FIG. 92.—The ova of the common human parasites (after Graham Brown).

*Tænia Solium.*

*Tænia Mediocanellata.*

*Ascaris Lumbricoides.*

*Tricocephalus Dispar.*

*Oxyuris Vermicularis.*

ject, which is a long one, must necessarily be curtailed here, the salient points of those which authorities are agreed should be regarded with apprehension, will be alone considered, and the following parasites, though occasionally present, are not important, viz., *Coccidia oviformes* (Leuckart), which infest most animals, and chiefly the liver and intes-

tines; *Cœnurus cerebralis* forms hydatids in the nervous system of the ox and sheep, it is the cystic worm of *T. Cœnurus* of the dog; *Cysticercus pisiformis* is found in the abdominal cavity and liver of the rabbit and hare; *Cysticercus tenuicollis* in the abdominal cavity of animals generally, *e.g.*, ox, sheep, pig, etc.; *Strongylus filaria* in the lungs of sheep; *Strongylus micrurus* in the lungs of cattle; and *Strongylus paradoxicus* in the lungs of the pig.

#### THE IMPORTANT PARASITES OF FLESH.

**Cysticerci.**—The cysticercus, or “bladder-worm,” causes the condition known as “measles” in the pig, ox, and sheep; and when the flesh thus affected is consumed, the “bladder-worm” undergoes a series of changes which terminate in its conversion into a *tapeworm*. In *the flesh of the pig*, and rarely in that of dogs, monkeys, or man, a number of small oval or round cysts are seen, occupying a position between the muscle fibres, and commonly varying in size from a pea to a cherry—though they have been found as small as  $\frac{1}{25}$  inch, and as large as  $\frac{3}{4}$  inch, in length. These cysts are the “*cysticerci cellulosa*,” *i.e.*, those bladder-worms which form a stage in the development of *Tænia solium*; they are surrounded by a pale milky looking fluid; and the cyst wall shows a white spot (generally central) upon its surface. The affected flesh is pale, soft, unduly moist and flabby, and it has a smooth slippery feel. Sometimes there is some degree of calcification of the capsule, and the result is that when sections are cut a grating sensation is experienced.

The bladders should be incised with a sharp knife, and the worm examined by a powerful hand-lens—when at one extremity will be found the blunt square head provided with a sucker at each “angle,” and a fringe of hooklets placed more

centrally ; these latter are very characteristic, and must always be found before a definite diagnosis is ventured on.

Those cysts that are dried up and indistinct can be made visible by soaking in weak acetic acid. Ostertag attaches



FIG. 93.—“ Measly ” Pork, showing (diagrammatically) its appearance to the naked eye.

great diagnostic importance to the rounded or oval calcareous corpuscles which are so generally embedded in the tissue of the head, but which disappear on the addition of acetic acid.

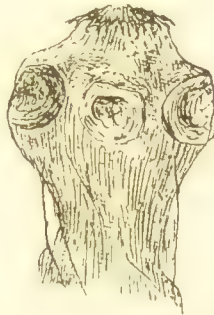


FIG. 94.—Head of *Tænia Solium*. (Obj.  $\frac{1}{2}$  inch.)

The younger pigs are especially attacked, and during life the earliest evidence of the parasites is afforded by the presence of one or more small cysts in the conjunctiva, or in the loose tissue of the frænum linguæ. After death the liver and the muscles of the shoulders, intercostals, and loins, are seen to be chiefly affected.

*The cysticercus of the ox.*—*Cysticercus bovis* or “beef-measles”—chiefly affects the calf ; it possesses a flat head armed with no hooklets, but simply suckers, around which



there is frequently a considerable deposit of pigment, and on the surface of the head there is a pit-like depression ("frontal suction cup"). It develops into the adult tape-worm called



FIG. 95.—Head of *Tænia Mediocanellata*. (Obj.  $\frac{1}{2}$  inch.)

*Tænia mediocanellata*, which is longer than *T. solium*, and appears to be more prevalent in this country.

***Bothriocephalus latus***, a tapeworm which is almost limited to certain parts of the Continent of Europe, is even larger than *T. mediocanellata*, has a club-shaped head, not armed with hooklets, but possessing two deeply grooved longitudinal suckers, one on each side.

***Tænia echinococcus*** is the small tapeworm, of 3 or 4 segments, which is commonly found in the dog. The encysted



FIG. 96.—Broad capsule of an *Echinococcus*.

stage ("hydatids") is most generally found in the lungs and liver of oxen, sheep and swine, and (more especially in Ice-

land) in man. The hydatids consist of thin pale vesicles floating in a clear liquid, and the whole is encysted in a tough capsule. The inner lining of the capsule consists of ciliated epithelium, and from the inside of the cyst wall there generally arise many so-called "brood capsules" (fig. 96).

The condition is diagnosed with certainty by the microscope, either by the discovery of the characteristic heads or of detached hooklets in the clear liquid of the cyst; valuable corroborative evidence is furnished by the fact that the liquid is quite free from albumin, and, in consequence, does not coagulate on boiling.

**Trichina spiralis.**—This parasite has been found in the flesh of many different animals (pigs, pigeons, eels, etc.), but most commonly, by far, in that of pigs; oxen and sheep do not suffer from attack by these nematodes.

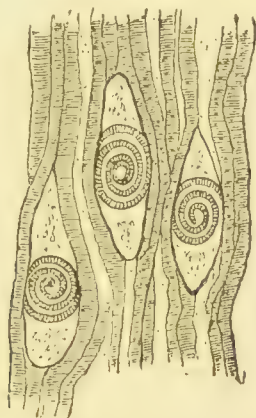


FIG. 97.—*Trichina Spiralis*, encysted in muscle. ( $\times$  about 40 diameters.)

The shape of the minute worms is nearly that of a typical nematode, *i.e.*, a slender rounded body tapers gradually at either end; the extremity which constitutes the head, goes to a long slender point which presents a small central orifice—the mouth; the other extremity, the tail, ends more bluntly. The worms possess a distinct alimentary canal, and even rudi-

mentary sexual organs are present; in the female an uterus is discernible, which will frequently be seen to be full of minute free embryos curved upon themselves; these latter have been observed to become extruded from the vagina, and subsequently to move sluggishly about the field of the microscope. The male worm is much smaller than the female, and is only about  $\frac{1}{16}$  inch long when mature; the latter reaches  $\frac{1}{8}$  inch. The long slender head and blunt tail are two characteristics which serve to distinguish these worms from parasites which otherwise resemble them, such as *Dracunculus* and *Filaria sanguinis hominis*.

The small worms are mostly coiled up in cysts, so disposed that their longest diameter is in a line with the muscular fibres, and a drop of acid will stimulate them to transient movements if they are alive. These cysts lie between the muscle fibrillæ, and their walls are sometimes partially or completely calcified—so as to give a grating sensation when the finger is passed over a section of the flesh. This calcareous deposit serves to shield the parasites from the destructive consequences of salting, and to a slight extent also from heat when the flesh is being cooked. There may be from one to three trichinæ in a cyst. Frequently 25 per cent. of these parasites are thus encysted in the diaphragm, and therefore, when possible, a piece of this muscle should be procured; the back muscles on the other hand are the least attacked.

Either a section may be made of the muscle, or it may be teased out with needles, and preferably, in the case of a long muscle, a point near its insertion should be selected—since this is a favourite site for encystment. The affected muscle is seen to be pale and œdematous, and if the worms are encapsuled, small rounded (or more truly, lemon-shaped), whitish specks, averaging about the size of a very small pin's head, are visible to the naked eye. These can be made very distinct by means of a hand lens; but a low power of the microscope should be employed in every case, and the most characteristic

appearance will be got by making a thin longitudinal section of the affected muscle, and immersing this in potassic hydrate solution of medium strength—which serves to make the muscle fibres transparent, and leaves the worm exposed in its coiled condition within the capsule. The soaking should not be prolonged beyond a minute or two, or the worm itself will also be cleared up. Glycerine is a good mounting medium when a permanent specimen is desired. Sometimes, owing to considerable calcareous deposit in and around the walls of the capsule, a view of the worm is obscured; in these cases a drop of dilute hydrochloric acid, run under the cover-glass, will dissolve up this deposit; or if, as is sometimes the case, an oil globule, or several, partially obscure the worm, a drop of ether, applied in a similar manner to the acid, will clear away the fat. There are generally oil globules at the poles of the capsule.

The parts which are most likely to be affected will easily be remembered if it be borne in mind that the worms migrate to their settlements from the gastro-intestinal tract, and chiefly from the commencement of the small intestine. The diaphragm, the liver, the intercostal and abdominal muscles, are necessarily the first encountered, and therefore suffer most; but in later stages of the infection there is rarely a muscle which may not be affected. It is also a common practice to make an effort to diagnose the presence of the parasites in the living animals, by examining the eyes and the under surface of the tongue, both of which will frequently show the small pin-head nodules.

The dangerous and often fatal condition created by these worms as they traverse the gastro-intestinal walls and travel to their encystment in the various organs of the body is most prevalent in those countries where the uncooked—or imperfectly cooked—flesh of the pig is consumed, as in the form of sausages, etc.

Hot smoking and efficient cooking destroy these parasites;

but in the latter case the meat must be "done through," *i.e.*, thoroughly cooked through the centre—or some of the parasites, especially when shielded by calcareous walls, may escape the temperature necessary to destroy them, *i.e.*, that of 150 deg. F.

There are small semi-transparent bodies called "Psoro-



FIG. 98.—One of Rainey's Capsules. ( $\times 285$ .)

spermia," or "Rainey's Capsules," which somewhat closely resemble trichinæ, presenting as they do small dark oval or elliptical bodies, of greater lengths than encysted trichinæ. They are, however, made up of a thick membrane formed by small hair-like fibres arranged in lines, which encloses small oval—or rather kidney-shaped—granular cells closely adherent together, and the whole lies embedded in the muscle substance itself, *i.e.*, the sarcolemma. They are extremely common, and may exist in the flesh of most of the animals used for human consumption, and apparently when eaten they do not harm the human being.

Several more obscure bodies, the nature and significance of which we are still more ignorant of, may exist in flesh; such as bodies somewhat resembling pus cells, and others forming minute concretions or tiny hard nodules. Interesting as these are pathologically, they are rare, and when present, even in numbers, do not appear to affect the wholesomeness of the meat to any degree.

**Actinomyces.**—The "ray-fungus" (*actinomyces*), one of the "fission-fungi," is now becoming recognised as a parasite of commoner occurrence in the ox than was once



suspected; the difficulties which stood in the way of an earlier appreciation of this fact, arose from the circumstance that both the ante- and post-mortem conditions of actinomycosis so closely simulate those of tuberculosis.

It has not yet been proved that the disease can be communicated by the flesh of animals (bovines) suffering from attack, and the vitality of the fungus when exposed to heat is very low. The subject is of such interest and importance, however, that a few additional facts are appended.

The parasite almost entirely affects the tongue, the jaws (especially the lower one), and the lungs, where they may be detected, by the naked eye, as small dirty white specks commonly about the size of a very small pea, but varying from the tiniest speck to  $\frac{1}{3}$  inch in diameter. The parasites assume, when encysted, a peculiar symmetrical appearance, due to the fact that they consist of small linear elements, thicker at one extremity than at the other, and are so arranged that their smaller extremities are all directed towards a central point; the stellate or rayed appearance thus created is sometimes remarkably regular and uniform. The tongue when affected is hard and swollen, and presents the flattened nodules chiefly upon its dorsal aspect.

**Distoma hepaticum.**—To examine for these parasitic trematodes, the liver should be taken, and the bile ducts



FIG. 99.—*Distoma hepaticum* (natural size).

carefully exposed. They will be found as small organisms of a pale brown colour, in shape like little soles, and provided at their broad extremity with a sucker for attachment to the



walls of the bile ducts. Their surfaces are beset with many little warty points, and they average in size from 1 to  $1\frac{1}{2}$  inches in length, and about  $\frac{1}{2}$  inch in width; they most generally attach themselves to the biliary ducts, but they may be found also in the parenchyma of the liver.

#### THE LIFE HISTORIES OF THE ANIMAL PARASITES OF MAN.

**Tænia solium.**—Portions of the ripe proglottides of the fully matured tapeworm are swallowed by pigs—or rarely by dogs, monkeys, or man; or, more commonly the ova they contain escape and become scattered—some into water, others upon grass or vegetables, where they may certainly survive for some days. If the eggs are ingested, on reaching the stomach, the shell gets dissolved by the gastric juice, and the embryo (a globular body armed with 3 pairs of hooklets) bores its way through the stomach or intestine, and finally comes to rest in some part of the body; it then grows in size, loses its six hooklets, and after a time develops a head provided with four suckers, and armed with a circle of minute hooklets (“bladder-worm,” or “cysticercus cellulosæ”). The head grows out from the inside of the bladder, to the wall of which it is attached by a constricted part known as the neck of pedicle. The parasite may remain in this condition for long periods, or may shrivel up and die, for it is incapable of further development until it is ingested by a carnivorous animal; when this occurs, on reaching the alimentary canal, it projects its head and neck (by invagination), the bladder part is dissolved by the gastric juice, and very shortly transverse lines appear on the neck, which increase in size and separate until after a few weeks a jointed adult tapeworm results, with proglottides charged with ova ready to commence a fresh cycle.

The life histories of **Tænia mediocanellata** and **Bothriocephalus latus** are similar to that of *T. solium*; but the

bladder-worm of the *Bothriocephalus latus* is supposed to inhabit some fish (perch, pike, and salmon-trout?)—or possibly a fresh water mollusc.

***Distoma hepaticum*.**—The ova develop, in water, into ciliated embryos, and these undergo in small water snails (*Limnæus truncatulus*) a further development to larvæ; these ultimately develop into little organisms resembling tadpoles (cercaria), which either remain encysted in water snails or leave them and become attached to grass. They are most generally taken up by grazing sheep, but very rarely man has also become a host.

***Tænia echinococcus*.**—Of the 3 or 4 segments of this tape-worm the last one only contains sexual organs. The ova are discharged with the fæces (commonly of dogs), and they probably infect oxen, sheep, swine and man through the medium of water or raw vegetables. On entering the stomach the gastric juice dissolves the shell of the ova, and liberates the embryos, which possess 6 hooklets in 2 rows; by means of these hooklets the embryo bores its way through the walls of the intestine and develops, chiefly within the liver, into so-called "hydatid cysts," *i.e.*, the hooklets are lost, and the formerly solid embryo swells out into a vesicle; generally a number of protrusions ("daughter cysts") grow from the interior of the vesicle, which itself forms a cyst ("mother cyst"), to which they are attached by a pedicle, which ultimately becomes detached. Each "daughter cyst" may develop "grand-daughter cysts," and thus the original echinococcus may become full of smaller cysts of varying sizes ("pill-box hydatids"). Finally little buds form, which develop into "brood capsules," *i.e.*, thin walled sacs which remain attached by a pedicle, each developing a number of heads, with 4 suckers and a row of hooklets.

Thus the encysted form of these parasites possesses the distinguishing feature of being able to give rise to a large

number of scolices, most of which are capable of developing into the adult worm when they enter another host.

Rarely the hydatid throws out protrusions externally.

**Ascaris lumbricoides.** (The round worm).—The ova of the females are discharged with the fæces of the host, and then they are capable of furnishing embryos, but not before ; these probably have an independent existence (possibly in water or some intermediate host—such as worms or insects) before again entering the human body and completing their development. The parasites inhabit the small intestine, are of a brownish-yellow colour, and are most commonly met with in people who live amid dirty surroundings.

**Oxyuris vermicularis.**—These fine, white, thread-like parasites occupy the large intestine. The ova, unlike those of *A. lumbricoides*, contain embryos prior to their discharge, but probably these are incapable of further development until they have passed with the fæces—when they may re-infect the same individual or others occupying the same bed, etc., or may pass into water, or get deposited upon vegetables and fruit, and thus get ingested.

The life histories of **Tricocephalus dispar** (whip-worm) and **Schlerostomum duodenale** (common in Egypt and Brazil) have not yet been definitely ascertained ; it is not yet certain in what vehicle the ova of the females (which develop in man) infect their host, or whether in either case there is an intermediary stage of development of the parasite.

**Bilharzia hæmatobia.**—The male is a white flattened worm, half an inch in length ; posteriorly the sides of the parasite curve towards each other, and meet to form a channel, in which the long slender female (three-quarters of an inch in length) lies during fecundation. The ova possess a beak, which generally projects from one end, but sometimes laterally ; these ova may be hatched before the parasite leaves the tissues of the original host, but the embryos are not born

until afterwards. If the ova find their way into water, their walls swell up, rupture, and the minute embryos escape, armed with cilia which serve to project them through the water. Probably the embryo becomes attached to some fresh water mollusc (or possibly fish) and develops into a cercaria form, is ingested as such, and then completes its cycle of development.

**Trichina spiralis.**—When the trichinous meat is consumed, the trichina embryos (averaging a little over 0.1 m.m. in length), which resemble small filariæ, bore their way through the intestines and reach the tissues. They always become encysted in muscle fibres, when they increase in size (up to 0.6 to 1 m.m. in length), and acquire an alimentary canal and sexual organs. The encysted worms remain quiescent for long periods, and may ultimately die, but if the flesh is eaten they give origin, through their embryos, to a fresh cycle of existence.

## FISH.

There are few points so easy to detect as commencing putridity in fish: this is fortunate, inasmuch as decomposition sets in rapidly, and appears to be more generally productive of poisonous symptoms than decomposing meat—the symptoms produced being very similar in both cases. The bright gills, the prominent eyes, the elastic resistance of the firmly adherent flesh, and the absence of any but the characteristic odour, all arise in evidence of freshness. The soft inelastic feel of the fish, and the unpleasant odour, furnish the chief clue—and the most reliable—of commencing decomposition, since some salesmen will revive the gills by artificial colouring agents, and have been known to keep the eyes prominent by a small piece of stick, fixed transversely in the head, so that it presses the eye outwards on either side.

Iridescence, and sometimes luminosity, is frequently seen upon the surface of the flesh of decomposing fish. Stale fish float, while fresh fish sink in water.

Fish are subject to parasitic attack, and notably is this the case with the cod, in which many varieties of these organisms have been found; cooking, however, effectually destroys them, and fortunately, in the case of fish, the flesh is not palatable unless the cooking is thorough.

The power of certain shell-fish to create at times, and with some persons, violent gastro-intestinal disturbance and urticaria is well recognised; mussels and oysters collected from near sewage outfalls have had virulent poisonous properties ascribed to them, and it is possible that they may convey the infection of enteric fever or cholera. Perch, turbot, pike, crabs, shrimps, salmon and sardines have all given rise to poisoning.

#### HORSE FLESH.

By the Horse Flesh Act, similar powers to those of the Public Health Act (1875) are given as to the inspection, examination, and seizure of horse flesh sold for human food, and which is not legibly labelled "Horse Flesh." It becomes necessary, therefore, in order to check fraud, to be familiar with the chief differences which exist between the meat of the ox, etc., and that of the horse. In horse flesh the meat is a darker red, and sometimes brownish; it is coarser—the muscular fasciculi being broader—than in ox flesh; the odour of the fresh meat is different, and after the lapse of a day or two, as the flesh dries, it develops a peculiar faint odour, and imparts a soapy feeling to the fingers. The fat is more yellow and soft, and possesses a sickly taste, and, in consequence, it is sometimes removed and replaced by ox-fat, which is skewered on to the meat. If the bones have not been removed they will afford an additional clue, inasmuch as they



are larger, and their extremities (tuberosities, etc., for the attachment of muscles and ligaments) are larger and more marked—in addition to some anatomical differences in the construction of the horse's skeleton.

Horse flesh is richer in glycogen than ordinary meat, and this is taken advantage of in the following test:—

A strongly concentrated infusion of the suspected meat is mixed, when cold, with dilute nitric acid in the proportion of 5 c.c. to 100 c.c. of the broth; it is then filtered and tested with hot (freshly-prepared) saturated solution of iodine, gently let down upon it so as not to mix the liquids. If the substance is horse flesh a marked red to violet ring appears at the junction of the two liquids.

The tongue and the liver of the horse, together with some other organs, are also occasionally made to do duty for the corresponding organs in the ox. The tongue of the horse is, however, broad and rounded at its free end, instead of pointed as in the ox; and, if the hyoid bone is attached, it is found to be made up of 5 parts—whereas that of the ox consists of 9. The epiglottis is, moreover, smaller and more pointed in the horse. The liver, whether of the ox or sheep, consists of one very large lobe and another relatively small one; in the horse there are three large and distinct lobes and a fourth which is very small.

**Sausages, pork-pies, meat-pies, brawn, etc.,** have all given rise to symptoms of poisoning (Welbeck, Nottingham, Arlford, Retford and Chester, etc.). The period which elapses before the symptoms supervene varies from 10—50 hours among those attacked.

The agent which creates these poisonous symptoms seems to have the power of appearing (and increasing) in meat which is undoubtedly good and healthy at the time when it is employed in the making of sausages, pies, etc.; for the fresh meat appears in no case to have caused any unpleasant symptoms. When, however, this meat is minced, cooked, and made up



into sausages, etc., and kept for a little, the poisonous agent may make itself manifest.

A toxin can only be separated by special and difficult methods, and identified by its action upon living animals. The subject is a very complex one, and its study is surrounded by many difficulties, but when one so frequently reads of poisoning by meat, milk, etc.—sometimes, alas! with a fatal result—and considers the many slight cases there must necessarily be where the food, though the offending cause, is not suspected of being so—the matter urgently calls for every effort to be made to extend our present knowledge, and to place the matter upon a more satisfactory and scientific basis.

In the present state of our knowledge it is generally believed that these poisonous elements are the products of bacteria, but all the conditions under which the bacteria appear and work are but imperfectly understood. In almost every case it transpires that the food has been prepared and kept in a raw state for at least a day or two prior to cooking; the meat, moreover, is generally of a kind and form especially liable to be insufficiently cooked, or eaten in a partially raw condition; and subsequent to imperfect cooking it has generally been stored for a varying period.

Probably the cause will be found as much in the miserable provisions now generally obtaining for meat storage as in anything, and when one considers the damp, dark, unventilated condition of larders generally, and their almost invariable proximity to the basement water closet or the dust-bin, it is a matter for surprise that there are not many more such cases of food poisoning. Cooking will not destroy these toxins when they are once formed; they are very resistant against heat.

Bacilli have been found by Klein in some cases only. In cases of doubt, the quickest and most direct means of ascer-

taining whether the meat is the offending cause is to give some to a cat or dog, and watch results.

It is not a difficult matter to detect early decomposition in **sausages**, since the alteration in the smell alone will generally suffice ; but if a little of the sausage is boiled with water, and some freshly-prepared lime-water is added, good meat yields only a faint ammoniacal smell, whereas bad will give off a peculiarly offensive ammoniacal odour. Putrefaction generally commences in the middle of the sausage.

The skins of sausages have been known to contain mineral poisons, but this is very rare.

Sausages are made of the chopped flesh or internal organs of various animals ; this is mixed with condiments, and filled into clean gut or parchment ; the sausages are then generally boiled, smoked, or scalded. Saltpetre is sometimes added to furnish a good red colour to the meat, and sometimes colouring agents and starch. The moisture should not exceed 70 per cent.

The best tests for bad and stale **eggs** are the following :—

1. Fresh eggs are most transparent towards their centres if held vertically against the light ; stale eggs are transparent at their upper extremities.

2. If two ounces of salt are dissolved in a pint of water, fresh eggs when placed in the solution sink, and stale ones float.

Toxic albumoses have been separated from decomposing eggs.

## CHAPTER IX.

ALCOHOLIC BEVERAGES, VINEGAR, MUSTARD, PEPPER, SUGAR.

## ALCOHOLIC BEVERAGES.

THE estimation of alcohol in alcoholic beverages is of importance, and as certain drinks have entered the market which are sold as free from alcohol, it is desirable to know how to proceed in order to ascertain if this is a fact.

The law allows an ample margin before regarding a beverage as an intoxicant—2 per cent. of alcohol being permitted.

Some of the liquid containing alcohol, or suspected of doing so, is measured out—say 300 c.c., and then placed in a retort and boiled gently, until at least 200 c.c. have distilled over into a flask. This 200 c.c. is next diluted to the original bulk of 300 c.c. with distilled water, and the specific gravity is taken in a specific gravity bottle, at the temperature of about 60 deg. F. If it is 1000 the fluid is free from alcohol, and the amount of alcohol which has distilled over will be great in proportion to the extent to which this specific gravity falls below 1000—since pure absolute alcohol at 60 deg. F. has a specific gravity of 793.8 (water at 60 deg. F. = 1000). To find the percentage amount of alcohol by volume which corresponds to the specific gravity found, it is useful to consult the excellent alcohol tables of Mr. Hehner or Dr. Stevenson, in which these data are arranged side by side, together with the percentage amount of “proof spirit” present.

**Spirits** constitute the distillates from various liquids containing alcohol; the spirit of commerce is obtained from malted barley, maize, or potatoes.

The expressions "over-proof," "proof," and "under-proof," have the following significance:—The term "proof" arose from a former mode of pouring the spirit over gunpowder, and setting fire to the vapour arising; if this fired the gunpowder, it was "over-proof"; if the spirit burned without igniting the powder, owing to the large amount of water left behind, it was "under-proof"; and the weakest spirit capable of firing the powder was called "proof"; such a spirit was stronger than the present "proof spirit."

By "proof-spirit" is now implied a mixture of 57.05 per cent., by volume, of pure absolute alcohol in water, having a specific gravity of 919.8 at 60 deg. F.; and solutions weaker or stronger than this are "under" or "over-proof."

The Sale of Foods and Drugs Amendment Act, 1879, fixes the following limits of the common "spirits." Brandy, whisky, and rum, may be 25 degrees "under-proof" (*i.e.*, the S.G. may be as high as 947.4 and there may be only 42.8 per cent. by volume, of absolute alcohol—corresponding to 75 per cent. of proof spirit).

Gin may be 35 degrees "under-proof" (*i.e.*, may only contain 37.1 per cent. by volume of absolute alcohol, and the S.G. may be as high as 956.3—corresponding to 65 per cent. of proof spirit).

The percentage amounts of absolute alcohol generally present in spirits = 40 to 60 per cent. Wines = 6 to 13 per cent. Strong ales and porter = 6 to 8 per cent. Small beer = 1 per cent.

"Fusel oil" is the most important impurity of spirit; it appears to be, bulk for bulk, more injurious than ordinary ethylic alcohol; it should not be permitted to exceed 0.02 per cent.

*Detection and estimation of fusel oil* (R. Röse's method).

—The process is based on the following principles :—

Chloroform possesses the property of rapidly removing amylic alcohol from its solution in diluted spirit, and the presence of amylic alcohol in chloroform increases its power to dissolve ethylic alcohol. When, therefore, chloroform is shaken with diluted ethylic alcohol containing amylic alcohol, there will be a notable increase in volume.

The apparatus required is a stoppered tube having the lower part drawn out and graduated l, and capable of holding about 180 c.c. ; the graduated portion holding about 50 c.c.

20 c.c. of chloroform are introduced into the bottom part of the tube by means of a long-necked funnel—so that it shall not collect on the upper sides of the tube. The spirit to be tested is first diluted or strengthened until it has a specific gravity of 934.3 at 60 deg. F. (*i.e.*, contains 50 per cent., by volume, of real alcohol) ; and 100 c.c. of this prepared spirit is carefully run on to the top of the chloroform. The stopper is greased with vaseline and tightly fitted, and the whole tube immersed for half an hour in water for the chloroform to settle (which process is aided by occasional tapping), and after an hour the volume of the bottom layer is read off. If the spirit is pure this volume will now be 37.1 c.c., but if it contains 1 per cent., by volume, of amylic alcohol, the bottom layer will measure 39.1 c.c., thus giving an increase of 1 c.c. for each half per cent. by volume of amylic alcohol present in the sample.

When this process is applied to the ordinary raw spirits of commerce the results are somewhat below the truth on account of the presence of other impurities which have less tendency to pass into the chloroform.

The presence of fusel oil may also be detected by (a) slowly distilling off the great bulk of the liquid and extracting the residue in the flask with ether ; the ethereal solution is



allowed to evaporate spontaneously, and then the residue is heated with sulphuric acid and sodium acetate when the odour of pear is evolved.

(b) Decolorising with animal charcoal, adding a few drops of hydrochloric acid, and afterwards some fresh and colourless aniline oil—in the presence of fusel oil the aniline compound acquires a rose tint.

In estimating alcohol the spirit should be first diluted with an equal volume of distilled water—when the alcohol found must of course be doubled.

The commonest form of adulteration is by the addition of water. When the distillate gives a decided red colour within 15 minutes, with 1 per cent. nitro-prusside of sodium solution and ammonia, methylated spirit is present. The senses of taste and smell alone afford an excellent index to purity.

**Beer.** — This beverage was formerly made from malt and hops only; now it can be legally made from starch and sugar, and various vegetable bitters.

From the malt the beer derives maltose, dextrine, albuminoids and salts, and from the hops a bitter principle, resin and tannin; alcohol, carbonic acid, a little glycerine and succinic acid are produced by the fermentation, and a little lactic and acetic acid results from schizomycetic fermentation.

With regard to the “finings,” which are added to clarify the beer or wine (and which are not regarded as constituting sophistication), their nature is very varying, and the detection of the composition of the “patent finings” commonly employed is a matter of the greatest difficulty, but isinglass and gelatine generally enter into their composition.

Calcium and sodium salts, ferrous sulphate, sulphuric, sulphurous, boracic, tartaric and salicylic acids, and alum, are the adulterants which have been employed. A solution of lead acetate will largely decolorise beer.

The adulterants here employed are all detected by methods



which will occur to the reader of the former pages of the book.

To test for salicylic acid the beer should be acidulated with sulphuric acid and well shaken with an equal amount of a mixture of ether and petroleum naphtha; let stand, and then pipette off the ethereal layer and evaporate down to a few c.c., add a little water and a few drops of dilute ferric chloride solution, and filter—when the filtrate, in the presence of salicylic acid, will be of a violet purple colour. Boric acid may be discovered in the ash by the procedure recommended in “Milk Analysis.”

The alkaline salts are added to correct undue acidity, but most of the prosecutions which have taken place under the Sale of Food and Drugs Act have been for an undue amount of common salt. Before it can be decided that salt has been added, allowance must be made for the salt in the brewing water, and for the chlorides natural to the malt and hops. Twenty grains of salt per gallon would be a most generous limit to allow on these accounts, but where possible the actual water used in the brewing should be tested for chlorine. The ash of beer should not exceed 0.5 per cent. Sulphurous acid may find its way into beer (and wine) from the practice of sulphuring the insides of the flasks, or washing them with a solution of calcium bicarbonate in order to destroy micro-organisms; but it is also added to regulate fermentation, and to produce a flavour of age.

Sodium bicarbonate has also been added to increase the effervescing property of the beverage.

Very rarely other bitters (quassia, calumba, gentian, and chiretta) have been used as substitutes for hops; this substitution has no public health import; noxious bitters have in former times been detected, such as picROTOXINE, nux vomica (strychnine) and picric acid, but these are not likely to be now employed.

The hop bitters are precipitated by neutral acetate of lead; if, therefore, a litre of beer is evaporated to about 300 c.c. and then precipitated while hot with a solution of neutral lead acetate, and the solution filtered and evaporated to 50 c.c., it has no bitter taste, whereas if hop substitutes have been employed the solution is distinctly bitter.

Probably the most important consideration connected with beer is that of its acidity, since an abnormal amount of this either denotes commencing changes of a deteriorating character, or implies the addition of sulphuric acid (employed to clarify, to lighten the colour, and to give the beer the hard taste which naturally only comes by age). The normal acidity of beer depends upon the presence of carbonic, acetic, lactic, malic, tannic, or gallic acids, and can be estimated in terms of *glacial acetic acid* by exactly neutralising it by decinormal soda solution (1 c.c. of which = 6 milligrammes of glacial acetic acid). The acidity of 100 c.c. of beer should not exceed 30 c.c. of decinormal alkali.

Before distilling for alcohol, the beer should be well shaken to expel as much carbonic acid as possible, slightly diluted with water, and rendered alkaline with caustic soda, and so as to keep the beer from frothing over, a small flame only must be applied to the flask, and a little tannin should be added.

Strychnine may be tested for in the manner indicated in Chapter XIII., but the beer must first be treated with animal charcoal and then filtered.

**Wine** is the fermented juice of the grape. It is much more adulterated than beer, and such adulteration is probably more generally and readily detected by the palate.

Here again the estimation of the acidity is of importance, but inasmuch as it varies considerably in the very numerous varieties of wines, the subject is a somewhat indefinite one. In this case, however, results are more generally returned in

terms of *crystallised tartaric acid per cent.*; 1 c.c. of the decinormal soda solution will neutralise 7.5 milligrammes of crystallised tartaric acid. The sample should be diluted before titration, and phenol-phthalein used as indicator.

If the acidity—which is chiefly due to such acids as tartaric, malic, acetic, formic, and butyric—exceeds 1.2 per cent. of

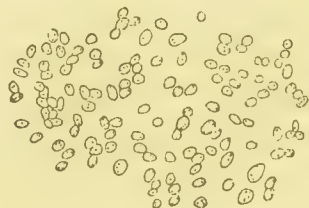


FIG. 100.—*Torula Cerevisiæ* (yeast plant). ( $\times$  about 200.)

tartaric acid, it is undesirable in a liquid of which considerable quantities are repeatedly drunk; and the sample is probably not a genuine one.

A microscopic examination, as in the case of beer, will sometimes detect fungoid, etc., organisms.

The commoner adulterants are water, sugar, various ethers, logwood, sulphate of lime and alum; and these are, of course, especially employed in the manufacture of the cheap clarets. Calcium sulphate is used to improve the appearance of cheap wines by furnishing a brighter and more permanent colour. This so-called "plastering" of wine is likely to be injurious to health; it is indicated when the  $\text{SO}_3$  in 100 c.c. exceeds 0.092 gramme; it gives rise to the formation of potassium sulphate, which has a decided purgative action; in France this salt is not permitted to exceed 0.2 per cent. Sodium chloride is sometimes added; it should not exceed 0.05 gramme in 100 c.c.

Astringent agents—commonly tannin, alum, and catechu—are also used.

In testing for salicylic acid in wines the same procedure as recommended in the case of beer may be adopted, but here it is well to re-acidulate the filtrate, then dilute, and

repeat the treatment with ether and petroleum naphtha before testing with the iron solution. Boric acid is said to be a normal constituent of wine, in small quantities (*i.e.*, about 0.02 gramme to the litre). It is best obtained by repeated distillation of the ash with small quantities of methylic alcohol, after moistening it with concentrated sulphuric acid.

Methods have already been given by which most of the adulterants can be detected:—The tannin in a measured quantity of wine may be estimated as in tea. In genuine samples of red wine the tannin does not exceed 0.25 per cent.

The chief colouring agents are logwood, blackberry, elderberry and prune juices, sandalwood, cochineal, magenta, Brazil wood, aniline reds and violets, and indigo; they are comparatively harmless; some of these colours are heightened in tone by tartaric acid.

“White” wines may be decolorised with animal charcoal, and “red” wines with basic acetate of lead and magnesium sulphate.

Many tests have been suggested for the detection of foreign colouring matter in wine, but few of them are found to be invariably satisfactory in practice; some of the best are:—

A 10 per cent. solution of good, clear gelatine is allowed to set, and from the firm mass several small cubes of about  $\frac{3}{4}$  inch square are cut; two or three of these cubes are immersed in the wine for 24 hours. In pure wines the colouring matter does not penetrate the gelatine for more than about  $\frac{1}{16}$  of an inch, but the majority of the foreign colouring matters penetrate almost, if not quite, to the centre of the cubes; dilute ammonia will dissolve out the colouring matter of cochineal and logwood, and will strike a blue with alkanet (Dupré).

(a) Baryta water is added to the wine until it acquires a greenish hue, and the wine is then shaken with acetic ether. The upper layer remains colourless, even after acidulation with acetic acid, if the wine is pure, but if *basic* coal-tar dyes are present, various characteristic colours result.

(b) Dilute potassic hydrate solution is added to a few c.c.'s of the wine, until the mixture is distinctly alkaline, then some mercuric acetate is added and the mixture shaken and filtered. The filtrate is colourless in the case of pure wines; but it is red or yellow if *acid* coal-tar derivatives are present (Preliminary tests employed at the Municipal Laboratory in Paris).

The colour of genuine wine does not dialyse to any decided extent, but that of logwood, cochineal and Brazil wood does so readily.

The presence of foreign colouring agents having been ascertained, it remains to discover their nature by special and appropriate tests. Some of these are indicated in Chapter XII., but the matter is in respect of some of the agents employed a difficult task; the reader is more especially referred to Part I., Vol. III., of *Commercial Organic Analysis*, by A. H. Allen.

Lammatine's suggestion serves to detect logwood and cochineal; it consists of shaking 100 parts of the wine with a large amount of coarsely powdered peroxide of manganese for a quarter of an hour, and then filtering through a double filter; if the wine is pure a colourless filtrate results; if impure the filtered liquid should be treated with zinc and hydrochloric acid, which destroy the coloration.

Wines are sometimes fortified with inferior brandies.

It may be necessary to examine any of these beverages for poisonous metals, in those cases where they are judged to have run the risk of such contamination—as by the use of lead and zinc utensils in their manufacture, the cleansing of bottles with shot, etc.

The darkening of wine which sometimes results from the presence of iron is not objectionable.



## VINEGAR (ACETIC ACID).

Vinegar is essentially dilute acetic acid—generally 4—5 per cent., along with a little acetic ether; vinegars made chiefly from unmalted barley, maize, rice, and other grains, and from sugar or molasses, are frequently sold as malt vinegar. Large percentages of pyroligneous acid (wood acid) are commonly employed; it is derived from the destructive distillation of wood, and generally a little caramel is added to make it resemble genuine malt vinegar. In this country very little wine vinegar is used.

Sulphuric acid is still sometimes added to vinegar; it undoubtedly increases any harm that may attend the use of this dietetic condiment, since the acidity is thereby increased, and insoluble salts of lime have a greater tendency to form in the body.

The acidity of good vinegar should never fall below 3 per cent. of real acetic acid (*i.e.*, glacial acetic acid,  $C_2H_4O_2$ ), and this fact, along with the specific gravity (which does not fall below 1.015 in good vinegar), will serve to detect the addition of water. The amount of sulphuric acid can be estimated, as in water, by precipitating barium sulphate. The precipitate is then collected, washed, dried, ignited and weighed, and the weight multiplied by 0.3436 gives the weight of  $SO_2$ .

Free sulphuric acid may be tested for by moistening a piece of cane sugar in the vinegar, and exposing to the heat of a water-bath. The residue is more or less carbonised if free sulphuric acid is present.

To estimate the total acidity of vinegar, take 10 c.c., dilute it with 90 c.c. of distilled water, and into 10 c.c. of this diluted vinegar (which will represent 1 c.c. of the undiluted liquid) run decinormal soda until the neutral stage is reached, using phenol-phthalein as indicator. The number of c.c. of soda



solution required  $\times 0.006 \times 100 =$  the percentage amount of acetic acid present.

In cases where vinegar has been added to "tinned articles," such as pickles, fish, etc., the liquid should be tested, where necessary, for lead, zinc, copper or tin, since the vinegar adds materially to the solvent action of the juices, etc., upon the vessels containing them. Copper can often be detected by allowing a piece of steel (as a knife blade) to lie in the liquid for a short time, when it becomes coated with a layer of copper.

The ash of vinegar, if alkaline, shows that no free mineral acid could have been present in the vinegar, and this is clear proof of its freedom from uncombined mineral acids. Such acids may be detected by adding 4—5 drops of a 0.1 per 1000 solution of methyl-violet; pure acetic acid vinegar shows no change of colour, but traces of free mineral acid causes the violet colour to change into a bluish-green or green.

Vinegar eels (*anguillula oxyphila*) do not appear to be injurious.

Genuine malt vinegar yields extractive matter to the amount of about 2.5 per cent.

#### MUSTARD.

The mustard in general use is practically a mixture of brown and white mustard seed ground to flour and mixed with a little colouring matter, other flour, and ground chillies; for the pure seed possesses too acrid a taste to be palatable.

None of the adulterants employed have a harmful nature; such are:—Turmeric, the different starches, and when much foreign material is added, a little cayenne pepper is frequently mixed in with the mustard in order to keep up its sharp flavour. All these are easily detected by the microscope. The brownish-red reaction of turmeric with ammonia,

and the bluing of starch in the presence of iodine, are both valuable and reliable chemical tests.

In several cases tried under the Food and Drugs Act, the Justices have held that the addition of flour is permissible



FIG. 101.—The cuticle of the *white mustard seed*. ( $\times 200$ .)

under the Act, as a preservative, for unmixed mustard does not keep well.

*White mustard* under the microscope presents certain well-marked characteristics. Foremost among these is that presented by the outer coat, or cuticle, which consists of a layer of large hexagonal, so-called "infundibuliform" cells, which present a central ostium occupied by other bodies—called mucilage cells; when water is added, these latter swell up, and escape from the mouth of the large hexagonal cells into the water, to which they appear to furnish mucilage.

Inside this layer are three less characteristic ones, the innermost consisting of a thin layer of large granular cells; and the interior of the seed comprises a fairly regular areolar network, containing granular matter and minute oil globules.

*Black mustard* is similar, but differs in the fact that the large hexagonal cells are absent.

#### PEPPER.

Pepper is considerably adulterated, but mostly with agents which are harmless:—Linseed, various starches (rice, potato, and pea-flours chiefly), the palm-nut powder, and the ground stones from olives, and shells of walnuts, etc. ("poivrete").

Any added mineral matter, as in flour, can be mostly

separated by shaking up thoroughly with chloroform. Sand or earthy matter generally gains access in the following manner:—

The berries are allowed to fall from the trees on to the ground, where many of their minute furrows become filled with the soil; or by the wearing of the stones between which they are ground the powder acquires some sand. A small margin has to be allowed for the presence of this *unavoidable* foreign matter.

According to the resolution of Bavarian chemists (1890) the limits of ash should be:—

For black pepper 6.5 per cent. (2 per cent. insoluble in HCl).

„ white „ 3.5 per cent. (1 per cent. „ „ ).

A rather rough, but serviceable method, recommended by Neuss, and quoted in Parkes' *Practical Hygiene*, consists in covering the pepper with concentrated hydrochloric acid, when, if the sample is pure pepper, it becomes of an intense and uniform yellow, and most of the foreign ingredients, remaining uncoloured, readily betray their presence. The microscope, however, furnishes the most generally useful means of detecting adulteration.

Unlike mustard, pepper contains starch.

*Microscopical characters of pepper.*—A transverse section of the *black pepper* berry shows the following notable



FIG. 102.—The cells forming the central part of the pepper berry.

( $\times 100$ .)

points:—Starting from the cortex, most externally is a layer two or three cells deep, arranged vertically, and very much resembling in appearance the bean-starch granules, *i.e.*, ovoid,

with a central linear hilum crossed by transverse markings; next follows an ill-defined layer of elongated cells arranged transversely to the foregoing, and then a sort of irregular reticular tissue containing oil globules; more internally still, a well-defined single layer of large vertical more or less flask-shaped cells appears. The rest of the interior of the berry consists of flattened angular cells, dovetailed into each other, and containing starch.

*White pepper* is simply the central part of the berry.

If pepper is adulterated with "poivre" the microscope discloses a large number of "stone cells."

The following is a chemical test for "poivre":—Digest for 48 hours 1 gramme of phloro-glucol in 50 c.c. of hydrochloric acid (S.G. 1.1), and then decant the clear solution. Just cover some of the pepper with this reagent and heat cautiously until fumes of hydrochloric acid begin to come away. "Poivre" furnishes a deep cherry-red colour, but pepper a yellow or faint brown.

"Long pepper" is very inferior in strength to other kinds; it consists of the fruit of *Chavica Roxburghii*.

#### SUGAR.

The sugar of commerce is almost entirely cane sugar—very little glucose coming into ordinary domestic employ; but many sugars are artificially coloured with aniline colours in minute quantities, and thus the superior and higher priced cane sugar may be largely replaced by beet sugar.

The colouring matter foreign to sugar may generally be detected by washing about 100 grammes of the sugar, in a flask, with alcohol (90 per cent.). If the dye is not removed, the washing must be repeated until it is. The solution is then filtered, evaporated to dryness, and again taken up in a little alcohol. A skein of wool slightly mordanted with aluminium acetate is placed in the solution, which is warmed

over the water-bath. The skein is removed after a while, washed with water, and dried, when it retains a permanent yellow dye. The colour natural to sugar will not furnish a solution which is capable of permanently dyeing wool.

In estimating the ash of sugar it is best to carefully ignite 5 grammes of the powdered sugar, mixed by means of a glass rod with 5—7 grammes of coarsely powdered quartz sand (previously ignited), in a platinum dish; the platinum dish is, however, perceptibly injured in the process. Any insoluble mineral adulteration would be detected by dissolving the sugar in water and then filtering. The ash of sugar does not exceed 2 per cent.



FIG. 103.—The sugar mite (*Acarus Sacchari*). Magnified.

The amount of sugar present in any substance is best estimated by Fehling's method, the rationale of which is:—If solutions of caustic potash and sulphate of copper be boiled together, the mixture becomes black—owing to the formation of the *black* oxide of copper; the presence of certain organic substances, however, and amongst them grape sugar, prevents the copper becoming so highly oxidised, and the *reddish-brown* sub-oxide of copper is in consequence formed. Cane, beet, and maple ( $C_{12} H_{22} O_{11}$ ) sugar have no such action, but by heating the clarified syrup in a water-bath along with dilute sulphuric acid for about 10 minutes, it is readily inverted into grape sugar.



By inversion, then, is understood the conversion of non-reducing carbo-hydrates into sugars of the formula  $C_6H_{12}O_6$ , which directly reduce Fehling's solution to a reddish-brown sub-oxide of copper.

**Fehling's solution** is made by dissolving 34.64 grammes of pure cupric sulphate in water, and diluting to 500 c.c. The solution so obtained is mixed with another solution prepared by dissolving 173 grammes of sodio-potassic tartrate in water, mixing this with 100 c.c. of sodic hydrate solution (sp. gr. 1.34), and diluting the mixture to 500 c.c. When these two solutions, each of 500 c.c., are united, we obtain 1 litre of ordinary *Fehling's solution*. 1 c.c. = 0.03464 gramme  $CuSO_4$  = 0.005 gramme of pure anhydrous grape sugar.

The solution should be preserved in small, well-stoppered bottles, kept full, and in the dark.

A known volume of this blue reagent is placed in a white porcelain dish and brought to the boiling-point, after which the solution containing sugar (made very dilute) is added by a burette, until the blue colour has quite disappeared and is replaced by a brownish-red one—due to the suboxide of copper. The amount of sugar required to reduce a given quantity of the Fehling's solution being known, the estimation of the amount of sugar in the liquid under examination is easily arrived at. Moreover, the precipitate of the suboxide may be collected, washed, dried and weighed, and taken as a direct estimate of the quantity of sugar which has precipitated it.

**Honey.** — The microscope, by revealing the absence of pollen grains, gives a certain indication of artificial comb prepared from paraffin wax, etc.



## CHAPTER X.

## COFFEE—COCOA—CHOCOLATE.

THE unground coffee bean, as it reaches the market, consists of the true substance of the bean more or less enclosed in a thin skin—which is always most evident in the furrow.

Dr. Bell gives an analysis of Mocha coffee which serves to show the respective amounts of the constituents of the raw bean, and the changes in these amounts which are brought about by roasting.

	Raw Coffee.	Roasted Coffee.
Caffein . . . . .	1'08	0'82
Saccharine matter . . . . .	9'55	0'43
Caffeic acids . . . . .	8'46	4'74
Extracted by alcohol and containing nitrogenous and colouring matter .	6'90	14'14
Fat and oil . . . . .	12'60	13'59
Legumin or albumin . . . . .	9'87	11'23
Dextrin . . . . .	0'87	1'24
Cellulose and insoluble colouring matter . . . . .	37'95	48'62
Ash . . . . .	3'74	4'56
Moisture . . . . .	8'98	0'63
	<hr/> 100'00	<hr/> 100'00

A hot water infusion of the ground coffee contains the oil, sugar, caffein, most of the mineral matters, dextrin, and some of the albuminous matters.

*Adulteration.*—Chief among the adulterants is **chicory**, which is prepared from the root of wild endive.

The sale of chicory is admissible, and its admixture with

coffee is now generally taken for granted—indeed the majority will not drink the coffee without chicory ; it only becomes an illegal and fraudulent adulteration when it is added to samples which are offered for sale as “pure” ; such, however, have now but a small place upon the market, and certainly would not constitute 10 per cent. of all the samples sold ; the admixture with chicory, etc., represents from 40 per cent. to over 90 per cent. in some samples. The substance is employed to blacken and thicken, and to give the slight bitter flavour so agreeable to “coffee” ; but, owing to the absence of the alkaloid, it has none of the refreshing and stimulating powers of the latter ; otherwise it has a very similar dietetic value.

There are several well-marked characteristics of chicory which serve to make detection of its presence an easy matter. To tabulate these :—

1. It has a peculiar and distinct odour, different to that of coffee.

2. A 10 per cent. infusion of dried chicory, made by well boiling with water and then filtering, is much blacker than that of coffee, and its specific gravity is higher, *i.e.*, rarely below 1018, and averages about 1024, while that of coffee never exceeds 1010, and averages about 1008.7. The taking of the specific gravity is also a test for the presence of some of the rarer adulterants of coffee, *viz.*, ground carrots, parsnips, turnips, and mangold wurzel—the infusions of which will all show a specific gravity of 1015 and over ; it is obvious, however, that these substances must be in considerable quantities along with the coffee to affect the specific gravity of the whole mixture very materially ; it is also manifest that from the specific gravities the extent of the adulteration can be estimated, upon principles which have already been dealt with.

3. Roasted chicory sinks in water *at once*, while roasted coffee floats for some time, and falls but slowly—due to the oil

in the coffee preventing the particles from being readily wetted; and in chicory the sediment in the cup is soft and pulpy, while coffee remains hard. Moreover, the particles of chicory become almost immediately enveloped in a light brown cloud, which, forming streaks in the water, quickly imparts a distinct brown colour to the whole. Coffee will take a much longer time to achieve a similar result, *i.e.*, from 15—20 minutes; and the other sweet roots with which it has been adulterated, *i.e.*, mangold wurzel, carrots, parsnips, etc., will require the lapse of several minutes. Sometimes a little oil is shaken up with the ground chicory which prevents it sinking; in these cases the oil can be extracted by ether, and the material then tested as to the readiness and extent to which it colours water, and as to the character of the sediment formed.

4. Microscopically, the characteristic cells and the dotted ducts of the chicory form a safe and rapid means of detection.

5. Other tests have been suggested depending upon the difference in the amounts of ash, silica, and sugar in the two materials, but these are not very serviceable. Coffee ash dissolves in water to about 80 per cent.; but the ash of roasted chicory to only 35 per cent.

Pure coffee ash is almost white; that from chicory contains more iron, and has a reddish tint.

6. The substitutes for coffee are practically devoid of caffeine (Determine as in tea).

7. A 5 per cent. infusion of pure coffee, precipitated with slight excess of basic lead acetate, yields a colourless supernatant liquid; but this is more or less coloured in the case of chicory (Albert Smith).

The percentage admixture of chicory with coffee might be approximately gauged by estimating the chlorine—which in pure coffee is only about 0.03 per cent., while in chicory it commonly amounts to as much as 0.25; it would be neces-

sary, however, to incinerate the coffee, and it would be impossible to completely effect this without *some* loss by volatilisation—which would be larger or smaller, according to the precautions and care taken by the operator.

Roasted beans, acorns, and other starches (potato, sago, etc.), are rarely employed, and when they are, a microscopic examination, together with the addition of iodine to the infusion (decoloured by animal charcoal), will readily detect them. An infusion of *pure* chicory is not blued by iodine, nor is a strained infusion of coffee.

Burnt sugar, or caramel, has been added to improve the colour, aroma, and taste of the infusion; it can be detected by the fact that it will very rapidly and deeply colour water; the shining particles of caramel stand out from the comparatively dull particles of the ground berry, and if the former are picked out with forceps, they will be found quite soluble. Artificial coffee beans have in recent years got upon the market; they are made from a paste of various starches, coloured and flavoured with a little coffee and chicory. Sometimes berries which have been exhausted by making coffee extract are stained and sold.

Sugar syrup has been employed to glaze exhausted beans, and also to cause the unused berries to retain their moisture.

To furnish an artificial glaze to roasted coffee, a liquid has occasionally been employed, which appears to be a very pure petroleum oil.

*Coffee extracts* are deficient in caffein and extractives.

### COCOA.

Cocoa nibs, on analysis, are found to mainly consist of the following ingredients:—

Fat—generally amounting to from 45 to 51 per cent.

Albuminous matter—commonly varying from 13 to 16 per cent.

An astringent substance resembling tannin.

Cellulose, starch, gum, cocoa-red (a substance of a resinous character, which furnishes the colour of cocoa).

Two alkaloids—"theobromine," which closely resembles the alkaloids of tea (thein) and coffee (caffein), and caffein—together amounting to about 1 per cent. (and varying from 0.5—1.7 per cent.).

Mineral ash (3.5 per cent.); and water (4.5—6 per cent.).

As much as about a half of the ash is soluble in cold water.

With regard to adulteration, the term has to be qualified with reference to cocoa very much as it is in the case of coffee. "Prepared cocoa" is mixed with sugar and starches (generally arrowroot or sago, sometimes potato), and these latter, by serving to disguise the large amount of fat, which otherwise disagrees with some persons, render it more generally preferred.

Some of the fat, however, can be readily removed from the nib by heat and pressure, and this plan is by far the preferable one, as the added starch does not, while it reduces the percentage of fat, provide a substance of equal nutritive value to the other constituents of the cocoa which it displaces. The microscope will at once detect the addition of such foreign starches by disclosing their characteristic granules.

The ground shells of the cocoa seeds are sometimes added as an adulteration.

The amount of fat, and of the ash which can be obtained from a cold water extract of the cocoa, furnish two tests of its purity. In the case of the ash this varies considerably in even pure samples.

The theobromine may be estimated by previously removing the fat and caffein by petroleum spirit, and extracting with chloroform for 4 hours in a Soxhlet; then evaporate off the chloroform, boil the residue several times with water, and to this add the water in which the fat has also been boiled;



evaporate to dryness in a platinum dish, and weigh the residue as pure theobromine (Diesing).

**Chocolate** is cocoa from which much of the fat has been removed; the paste remaining is then mixed up with a considerable quantity of sugar and flavouring substances.

**Microscopical characters.**—The microscopical characters of the raw *coffee* berry are distinctive. The testa or skin—portions of which are always ground up with the rest—consists of long spindle cells with tapering rounded extremities, which are dovetailed into each other. Their characters, by the  $\frac{1}{4}$  inch power, are well shown in the accompanying figure. The internal substance of the berry is made up of

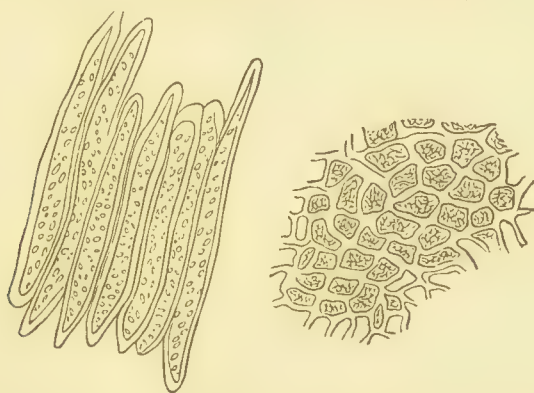


FIG. 104.—Coffee. Cells of testa and cellular structure. ( $\times$  about 200.)

a thick areolar tissue, the meshes of which are very irregular in size and shape, and contain, in addition to starch, yellow

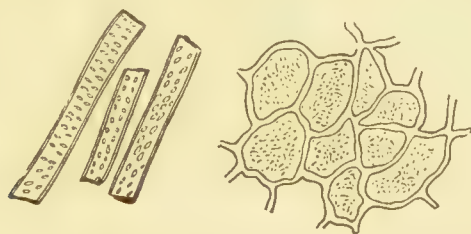


FIG. 105.—Chicory. Dotted ducts and cellular structure. ( $\times$  about 200)

angular masses and one or more rather large oil globules; the walls of this meshwork are somewhat beaded in appearance.



In *chicory*, the cellular tissue, the large dotted ducts, and the lactiferous vessels are characteristic.

The cellular tissue consists of an oval or rounded mesh-work, and is coarser than that of coffee. The lactiferous tubes are long pale and narrow branching tubes, which are

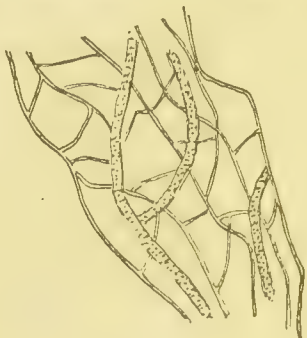


FIG. 106.—Lacteal vessels of chicory.

filled with a substance called "latex." The dotted ducts appear as jointed tubes marked with bars, and possessing square extremities.

Of the other (and rare) vegetable adulterants of coffee, *mangold wurzel* possesses a cellular tissue which has a tendency to consist of larger and more distinct elements; the dotted ducts have fewer joints, there are no lactiferous tubes, nor is there any starch.



FIG. 107.—Cocoa Starch Cells. ( $\times$  about 200.)

*Turnip* closely resembles the former, but the presence of a considerable amount of woody tissue serves to distinguish it. It likewise contains no starch.

*Carrots and parsnips* contain starch, but no lactiferous tubes.

Microscopically the small starch granules of *cocoa-nibs* are frequently seen to be massed together in the cellular areolar tissue, which, under a high power, is seen to be hexagonal.

The most external layer of the husk of the cocoa bean consists of long flat quadrangular cells; but the bulk is made up of large distinct and rounded mucilage cells.

## CHAPTER XI.

## TEA—INFANTS' FOODS.

## TEA.

THE leaves of the tea-plant (*Thea sinensis*), as they find their way into our markets, are generally mixed with some of the flower-buds, together with numerous small stems of the plant. The difference between black and green teas is entirely due to their method of preparation; they are both derived from the same plant.

An average sample of Black Tea shows about the following percentages of the two most important and characteristic constituents:—

Tannin, 12—13 per cent.

Thein, 3—4 per cent.

The ash is about 6 per cent. or a trifle over, and moisture commonly amounts to 9 per cent.

In the case of Green Tea, the thein and moisture appear to be generally less and the tannin considerably more.

Thein is the alkaloid of tea, to which it gives many of its most valued properties; the substance has been estimated as high as 6 per cent. in some samples. The caffein of coffee and the thein of tea are apparently the same substance.

The other constituents of the tea-leaf are:—Cellulose, vegetable albumin, extractives (by alcohol), chlorophyll and resin, pectin and pectic acid, dextrin or gum (Bell).

An infusion of tea leaves should be made by boiling 2 grammes of powdered tea in 100 c.c. of water for one hour; filtering while hot, and repeating until no more colouring matter is extracted.

An infusion of the leaves will be found to contain the dextrin or gum, tannin, thein, most of the salts, and some of the albuminous substances, pectin, etc. A good judge of tea will form a ready and approximately accurate estimation of its purity and genuineness by the smell and taste of a fresh infusion.

It does not appear, at first sight, an easy matter for adulteration to be practised in this connection, and yet formerly there were probably few articles of commerce less systematically exposed to fraud. Fortunately this fraud—which is generally perpetrated before the leaves reach this country—

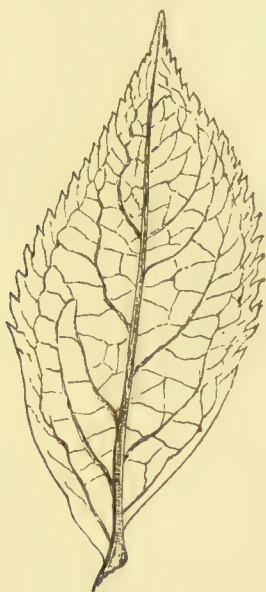


FIG. 108.

The elder leaf (after Bell).

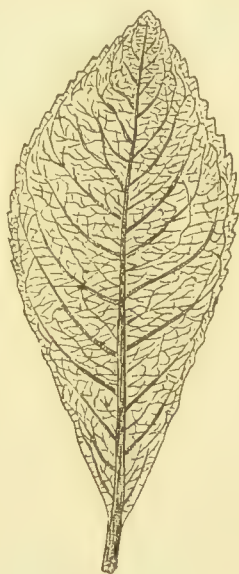


FIG. 109.

The willow leaf (after Bell).

rarely takes a very objectionable form hygienically, and has of late years very much diminished.

The admixture with foreign leaves is very rare indeed, but the addition of leaves which have been already infused is a form of sophistication which has been much practised. How can the former practice be best detected? The microscope

affords the readiest and truest means. A very low power will suffice to disclose the structural characters of the various leaves, and fortunately the tea leaf possesses characteristics which mark it out from those of any other plant; the differences, however, are slight, and may be readily over-looked, especially when the leaves of the elder, willow, and sloe—which are those that have been most commonly employed—are selected to serve the purposes of sophistication; there is much less difficulty in detecting the presence of beech and oak leaves when these are added.



FIG. 110.  
The sloe leaf (after Bell).

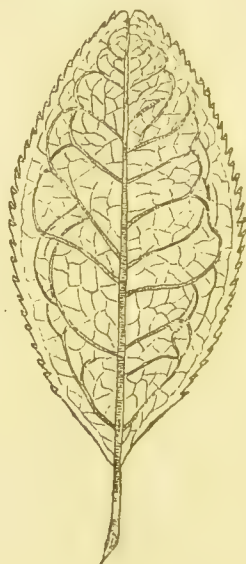


FIG. 111.  
The tea leaf (after Bell).

The common method of examination is to soak the leaves in warm water, to spread them out between glass slides, and then, by holding them up to the light, all the chief characters, including the venation, can commonly be discerned—more especially if a hand lens be employed; it is commonly necessary, however, to proceed to a low power of the microscope before a definite conclusion can be arrived at.

It assists in the detection of the characteristic structure of the leaves if they are previously heated with a solution of sodium hypobromite.

The characters of the *tea* leaf, when thus examined, are these:—The shape is elliptical; it averages from about one to two inches in length, and from half to one inch in breadth. The margin of the leaf shows distinct serrations, each of which is surmounted by a small spine, and these serrations do not quite extend to the point of attachment of the stalk; the apex is slightly emarginate; the primary veins come off dichrotomously from the midrib, and then branching off, form a markedly looped network extending to near the margin of the leaf, where, by bending back, they leave a narrow clear space.



FIG. 112.—The epidermis of the under surface of the tea leaf. ( $\times 285$ .)

Under the microscope the leaf shows an epidermic layer of flattened cells possessing well-marked sinuous outlines; coming off from a few of these cells are long slender unicellular hairs. On the under surface of the leaf there are a great number of oval stomata visible (fig. 112).

The most distinguishing characteristics of the tea leaf are:—The spine mounted serrations, which terminate a little before the point of attachment of the stalk; the looped venation; the notched apex; the long slender unicellular hairs; and the large number of stomata upon its under surface.

The leaves of the elder, willow, and sloe, are shown in



figs. 108, 109, and 110, in order that they may be compared; the difference will be seen to be so slight that their early and extensive employment as adulterants will be readily accounted for.

The leaf of the *elder* is seen to differ in the following respects:—Its shape is ovate; the margin is sharply dented, and the apex is pointed. The leaf is, moreover, seen to be asymmetrical—due to the fact that one lateral half of it is attached lower down the midrib than the other.

Microscopically, the epidermic layer of cells upon the upper surface of the leaf are less sinuous than in tea, and the cells present marked striæ. The unicellular hairs are either short, thick and conical, or short with bulbous free extremities.

The leaf of the *willow* differs in the following respects:—It is elliptico-lanceolate in shape, with a pointed apex, and its contour presents shallow serrations.

Microscopically, the epidermic layer of cells is smaller, their contour is not sinuous, and the unicellular hairs are long and tortuous.

In the leaf of the *sloe* the shape is similar to that of the willow, and the margin is slightly more deeply serrated than that of the tea leaf.

Microscopically, the epidermic layer of cells are smaller, their contour is not sinuous, and those on the upper surface of the leaf are striated. The unicellular hairs are shorter and thicker at the base than those of tea, and upon the under surface of the leaf the stomata are seen to be far smaller and fewer in number.

The question as to how the employment of used leaves may be detected is a much more difficult one to answer, for they may be prepared to resemble those which are unexhausted. By uniformly colouring or "facing" them, in the case of green teas, with a mixture of Prussian blue, turmeric, and sulphate of lime, and in the case of black teas, with black

lead—their appearance may be made to closely resemble that of unused leaves ; but this is now rarely, if ever, done. The natural perfume is sometimes added to, and apparently harmlessly so, by packing the leaves with certain aromatic flowers, which are generally removed before the tea is placed upon the market ; and catechu may be added so as to strengthen the infusion. The old leaves have been rerolled and worked up with sand and gum ; the sand or quartz serves the purpose of furnishing the stiffness of the natural unexhausted leaf, when dried ; and the gum, while also aiding in this, imparts a gloss to the otherwise dull looking leaf.

The task of detecting leaves which have been already used, is well nigh impossible to achieve, when these are only added in reasonable quantity, for genuine samples of tea vary so much as to the relative amounts of their constituents, that leaves of some samples may even have been partially exhausted, and yet yield more extract to boiling water, etc., than a very poor, but genuine sample.

Most will probably be learnt of this form of adulteration by the analysis of the ash of the leaves, and Dr. Bell found, that in average samples of genuine teas (dried at 100) this never reached 8 per cent. (generally from 4.7—6.2 per cent.)—except in one sample out of a considerable number, and the Society of Public Analysts has adopted this as the limit ; and in only one case did the ash soluble in water fall below 3 per cent. In America it is held that the soluble ash should not fall below 40 per cent. of the whole ash ; in this country the low limit accepted is 30 per cent. It follows, of necessity, that a sample of tea containing much exhausted leaf, will show a reduction below the limit of the soluble ash (frequently below 3 per cent.) ; this should be estimated by taking a weighed quantity of tea (say 10 grammes), gently incinerating this in a platinum dish, and weighing the ash—which is generally grey or greenish in colour. In faced teas

the ash is sometimes 10 per cent., and in "lie-tea" it may amount to 30 per cent. The ash should then be treated with boiling water, and the whole filtered through a small Swedish filter paper; the insoluble part must next be thoroughly washed on the filter, re-ignited, and weighed—the difference between the weight obtained and that of the total ash will be due to the loss of soluble matter. If the insoluble residue is boiled with dilute hydrochloric acid, that which still remains insoluble will consist mostly of sand and quartz—which should not much exceed one per cent., or the sample is not pure.

The estimation of thein is also of value where the use of exhausted leaves is suspected. Five grammes of the finely powdered tea are three times extracted with about 300 c.c. of boiling water, each extraction occupying one hour; the united extracts are then precipitated with neutral lead acetate; the filtrate is freed from lead by sulphuretted hydrogen, evaporated to dryness on the water-bath with freshly ignited magnesia and clean coarse sand, and the dry residue (finely powdered) is thoroughly extracted with chloroform in Soxhlet's apparatus. The residue by evaporating the chloroform extract is boiled with water, filtered, the filtrate evaporated down, and the residue dried at a temperature not exceeding 212 deg. F., and weighed. Under the microscope the thein appears as long white silky needles.

It is of great importance that the preliminary extraction is made with the greatest thoroughness, on account of the obstinacy with which a little of the alkaloid is apt to be retained by the vegetable tissue.

Dvorkovitch, when estimating thein in tea, washes the extract with petroleum ether in order to remove the oil and any traces of the brown substance found in tea.

Sodium carbonate and borax have been added to deepen the colour of the infusion.

The presence of catechu may be detected, except when it is in very small amount, by precipitating an infusion of tea leaves by a little solution of neutral lead acetate, then filtering, and treating the filtrate with a drop or two of a dilute solution of ferric chloride. A bright green colour appears, in the presence of catechu, which ultimately settles as a precipitate of a more sombre hue.

The tannin may be roughly estimated from the infusion of a weighed quantity of tea, from which it may be precipitated by gelatine—40 per cent. of the precipitate, when collected and dried, will consist of tannin.

The precise estimation is difficult and unsatisfactory. Löwenthal's process, as modified by Proctor (*Journal of the Society of Chemical Industries*, iii., 82, and v., 79), is the best. In this method it is ascertained how much permanganate of potash is reduced by tannic acid, and other readily oxidisable substances in the infusion; the available tannin is then precipitated by gelatine, and then it is seen how much permanganate is used up, the difference representing the amount of permanganate decomposed by tannin.

"Lie-tea" is the name given to a mixture of tea-dust with other leaves, clay, sand, etc., and made up into small masses with gum or starch.

#### INFANTS' FOODS.

There are many proprietary articles upon the market which have a large sale as "Infants' Foods"; and they vary considerably in their value as nutrients. Condensed milk has perhaps the largest sale; it consists of either cows' or goats' milk, sweetened with sugar, and then evaporated down to a syrupy consistence. Condensed milk contains generally about 10 per cent. of fat, that is to say, only a little over three times the amount in human milk. There is no limit fixed as to the amount of fat which such preparations should contain,

but considering the extent to which they are diluted when given to infants, it certainly ought not to be, in any instance, less than 10 per cent.

To estimate the fat the milk should be previously well diluted with about 10 per cent. of water, and then dealt with—preferably by Adam's process.

It is of great importance that starch shall not enter into the composition of infants' foods, for the reason that infants are incapable of properly digesting it. The presence of starch may be best ascertained by the microscope, especially if a drop of iodine solution is allowed to run under the cover glass. The nature of the starch employed will also be detected by the microscopic examination. A quantitative estimation may be made by extracting the soluble matter from the preparation by cold water, and drying the residue at a low temperature; an aliquot part of this is then inverted, and the amount of starch calculated from the amount of sugar thus obtained (as estimated by Fehling's solution; 1 c.c. = 0.0045 gramme of starch, after conversion).

There should be phosphates in these preparations; they may be dissolved out from the ash by nitric acid, and estimated by the method employed in water analysis.

The total nitrogen may be estimated by Kjeldahl's process.

The "humanised milk" preparations now sold are generally skilfully prepared and reliable, and doubtless afford the best artificial food that can be given for the first nine months of human life.



## CHAPTER XII.

PRESERVED AND TINNED PROVISIONS—COLOURING AGENTS  
EMPLOYED IN FOOD.

MOST articles of food, when preserved in "tins," run some risk of contamination, and the source from which this danger issues is in either one of the following directions.

1. Changes in the food itself—generally of decomposition, owing to preservative precautions being inefficiently taken.

2. Substances added—as antiseptics—to preserve the contents.

3. Substances employed as colouring agents.

4. Impurities yielded by "tins," or the solder used in their manufacture. By the action of the juices upon the "tins," arsenic, copper, lead, tin, or even zinc, may thus be taken up from the "tin" and solder. It is the vegetable, etc., acids naturally in the food, those which are added to it, or those that are formed during fermentation of vegetable matter, and oily substances, that act chiefly upon the "tins," and this action is increased by the galvanism which is sometimes set up between the metals present. Tinned asparagus is especially liable to take up metals, doubtless owing to the aspartic acid formed.

The most important consideration under the first heading arises from the fact that in some preserved meats (bacon, sausages, sardines, minced beef, etc.), poisonous products.



under certain ill-understood conditions, are apt to form, and such foods give rise to symptoms of a choleraic type among those partaking of them.

No definite and characteristic pathogenic organism has been described, and the poison is generally considered to be a chemical poison elaborated by micro-organisms. Such poisons are believed to be produced by the action of putrefactive bacteria upon the proteid constituents of the food, and toxic albumoses and crystallisable animal alkaloidal substances result; these may either be present in the food when consumed, or may develop after the introduction of the food into the body, and probably this accounts for the difference noted by the incubation periods in some outbreaks.

The potted meat, sardines, etc., are generally to all appearances sound.

The contents of the "tins" in preserved articles of diet are hermetically sealed down by solder at the boiling temperature, and the partial vacuum thus created in the "tins" is evidenced by their tops and bottoms being slightly depressed from the outside; should, however, there be any flaw in the "tins," or the solder seal be imperfectly applied, or should the heating process be but partially performed, then the contents may go bad—and in the latter case, owing to the accumulation of the gases of putrefaction, the tops and bottoms of the "tins" become quite flat, and later on, convex outwards, and the tin when struck gives out a hollow or drum-like sound. It is not difficult, therefore, in the majority of cases, to detect, before opening them, those "tins" in which the contents are bad.

By thus raising the contents to the boiling temperature, and then hermetically sealing them, those micro-organisms which induce decomposition are destroyed, and their further access is guarded against; nothing is thereby added to the food article, and thus this process, along with such methods of

preserving as freezing and sun-drying, are undoubtedly the safest and the best.

As a means to the same end, the employment of agents which will prevent the development of micro-organisms, and termed "antiseptics," is extensively practised. There seems little doubt that the use of some of these should be condemned, for although in the case of those more commonly employed their use has not been *proved* to cause any direct harm to consumers, it is rational to believe that such a salt as sodium salicylate may effect slight and indirect injury to health, and it is known that boracic acid deteriorates the blood and interferes with digestion.

Few of these agents enter normally into the constitution of the human body, and at least they must be regarded as foreign bodies whose ingestion works no possible good, and which, not being foods, do not in any way make amends for the additional work of elimination which their presence demands.

They permit, moreover, the vendor or manufacturer to deal with stale or badly prepared food. If he is permitted to practise such adulteration, at least he should be compelled to state the nature and amount of preservative employed.

The antiseptics most commonly employed are:—Borax and boracic acid, salicylate of soda, salicylic acid, benzoates and benzoic acid, sodium chloride, and vinegar; but salt-petre, chloride of ammonium, the sulphates of potassium, sodium and calcium, alum, creosote (which furnishes a smoky flavour), spirits of wine, sulphurous acid, bisulphite of lime, sulphate of copper—have all been employed.

Formaldehyde (known to the trade as "formalin") is also used as a preservative.

In order to preserve joints they have been coated with collodion, solid paraffin, and a mixture of gelatine and charcoal, and antiseptic agents have been injected by fine syringes into the flesh.

Some continental investigators have traced poisonous symptoms to the presence of tin. A tin sulphide is formed by the action of the albuminous matters upon the receptacles made of that metal, and it would be well to prohibit the employment of tin plate unless it be coated by some special preparation. Varnished tins are used by some French manufacturers, and lead has sometimes been found in this varnish. Tin is slightly soluble in acetic and other organic acids, and readily so in hydrochloric acid, but its absorption is very slight in the gastro-intestinal canal.

The acidity necessary for the juices and liquors of "tinned" and potted articles in order that they shall take up metals from the pots or from the solder used to seal them, is generally furnished by vinegar (acetic acid)—as in pickles, fish, etc.; and where this acid is employed there is always a special risk of metallic impurity being taken up; hence these are the cases where the liquid contents should be most generally tested.

It is not necessary to repeat here the means of testing for the various metals, since their characteristic reactions have been seen in treating of "Water." The presence of copper, however, can often be roughly demonstrated by allowing a piece of steel, such as a knife-blade, to lie in the liquid for a short time, and noting the appearance of a bronze coloration upon it. To extract copper from the ash of potted food, the grey ash should be treated with a little concentrated sulphuric acid, and then the residual carbon burnt off in a muffle. The ash should then be extracted with nitro-hydrochloric acid.

Having in view the fact that aluminium is now becoming cheaper, and that the metal is so extremely well adapted for making into "tins," pots, canteens, etc. (on account of its lightness, ductility and malleability, and owing to the fact that its bright appearance is very little affected by damp), the subject is growing into importance. The results of recent

investigations do not yet appear to be conclusive on all points, but alcohol can dissolve up the metal in a slight degree, and acids—even acetic and lactic—have a similar power, though much more marked. It is probable, however, that even in the latter case there is not sufficient dissolved to give rise to symptoms of poisoning under the common conditions of food-potting—for the metal is not a very poisonous one.

The tin-foil around sweets, etc., sometimes furnishes lead, and glazed and enamelled vessels may also do so. Doubtless many of the cases of poisoning that are attributed to metals are really the results of ptomaines, etc., in bad food.

#### THE COLOURING AGENTS EMPLOYED IN CONFECTIONERIES, PRESERVED FRUITS AND VEGETABLES, AND AS "DYES," ETC.

The only important matter in this connection, from a health standpoint, is to examine these various articles so as to ascertain whether the agents employed for colouring purposes are of a harmful nature or not.

The study of tinctorial chemistry is a special and a difficult one, but the health student must know of what the agents employed for colouring purposes may consist, and how to proceed in order to satisfy himself whether they are harmful or not.

1. **The harmful colouring agents** are for the most part of mineral origin; and the list which follows comprises those which are most likely to be met with.

*Lead* has been employed for the production of yellow, red and white tints.

*Arsenic* for yellow, orange, magenta, violet-lake, and (in the form of Scheele's green, *i.e.*, the arsenite of copper, or Schweinfurth green, *i.e.*, the aceto-arsenite of copper) green.

*Copper* for green and blue.

*Zinc, cadmium, tin and barium* for white.

*Chromium* for yellow and green.

*Cobalt and manganese* for pink and blue.

*Mercury* for vermilion-red.

*Antimony* for yellow and orange.

*Iron* for various shades of brown, and for the colour known as Prussian blue. The colour of Prussian blue is discharged by potash solution and restored again by acids.

*Gamboge and picric acid* for yellow.

Gamboge is very insoluble in water, but readily so in alcohol. Its presence is detected by dissolving out the colouring matter from the article by alcohol, filtering, and then, when water is added, the gamboge resin is precipitated; this precipitate will dissolve in ammonia with the production of a blood-red colour.

Picric acid (tri-nitro-phenol) can be extracted by alcohol and ether; it is detected by drying on the water-bath after the addition of a solution of the cyanide of potassium and caustic potash, when the blood-red colour of the iso-purpurate of potassium forms.

*Aniline* colours are liable to contain arsenic, as for example in the production of rose aniline, arsenic acid is used as an oxidising agent; but if tested and found to be free from this metal they are mostly harmless; some, however, such as naphthol green, aniline yellow, Martius' yellow, Bismarck brown, methylene blue, and gentian violet are more or less poisonous, apart from the presence of arsenic. As a rule only minute quantities of these aniline colours are employed at a time.

Aniline colours may be detected by the iso-nitril test:—To a little of the extract an equal amount of potash-lye is added, followed by 2 drops of chloroform; if the whole is gently warmed for a minute, and then boiled, the characteristic smell of iso-nitril generally becomes perceptible. The



well-known bleaching solution reaction for aniline can be readily got by adding an excess of the reagent to a solution of aniline in water.

2. The various **harmless colouring agents** are very numerous, and are almost all of animal and vegetable origin. To enumerate those which are most commonly employed in the production of various tints:—

A *red, pink, crimson or lake colour* is generally produced by either *cochineal* or the "*aniline reds*" (fuchsine and magenta).

The former colouring agent is derived from the cochineal insect, and is the more commonly employed. It can be detected by its character of turning violet with alkalies, and yellowish-red with acids.

The reddish colours extracted from the root of the *madder*, from *beetroot*, and from *safflower*, are also used.

The colour due to safflower turns light brown, and bleaches when treated with concentrated sulphuric acid.

*Logwood* is also of common employ, and may be tested for by extracting with alcohol, and then adding alum and ammonium carbonate—when a lavender or blue colour results (*vide* Bread).

A *yellow, amber, or orange hue*, is commonly imparted by *annatto*, *turmeric*, *aniline-orange*, *marigold* (the extract of which yields a permanent dark olive green with concentrated sulphuric acid), *chrysophanic acid* (which is extracted from rhubarb, and yields a fine purple colour with potash), and *saffron*.

To distinguish between the comparatively harmless aniline-orange (Victoria yellow—which is much used) and picric acid, evaporate off the spirit by which the colour was extracted, and cautiously taste for the bitter flavour of picric acid; then treat with a little dilute hydrochloric acid, and



when the solution has become nearly decolorised, introduce a piece of zinc for about an hour; picric acid yields a fine blue, and Victoria yellow a blood-red.

Annatto is very extensively used as a colouring agent, and like turmeric and saffron it is readily soluble in alcohol, though not in water; if in either case the alcohol is subsequently evaporated down and touched with a drop of concentrated sulphuric acid, annatto and saffron turn dark blue with a greenish tint, which in the case of saffron changes to a reddish-brown, and saffron furnishes a very peculiar odour; turmeric yields a violet red, and will turn brown with alkalies.

Annatto is obtained from the seed of a plant named *Bixa orellana*, the starch grains of which, though smaller in size, closely resemble those of bean. While it is at the present day one of the most extensively employed adulterants, it is itself subjected to considerable adulteration—turmeric and the flours of the various cereals being most frequently added, together with calcium sulphate and carbonate, Venetian red, etc. Copper is also sometimes added to prevent the growth of fungi. A microscopic examination, followed (in the case of the minerals) by a chemical analysis of the ash, will be required to detect the presence of these various adulterants.

Martius yellow gives a golden yellow solution from which acids separate a white precipitate; this is not the case with picric acid.

*A violet or blue colour* is frequently derived from the use of the *aniline* blues and violets.

Methylene-blue may be detected by adding hydrochloric acid to the extract, when a greenish precipitate results. Zinc dust reduces it, but the colour returns after exposure to the air. It contains zinc.

Methyl-violet extracted and treated with hydrochloric acid yields first a green and then a yellow colour.

*Indigo* is extensively employed, and it sublimes in dense violet vapours when the article is burned. The colour is discharged by the permanganate of potassium in the presence of potash solution.

The blue colour of *litmus* affords another means of such coloration. This substance is derived from *Rocella tinctoria* and from certain other lichens.

A purple colour is usually formed from a mixture of blue with some vegetable pink, such as rose-pink, logwood and cochineal.

The various shades of brown are most commonly imparted by heating sugar to various stages ("caramel"), and a green colour is now almost invariably obtained by the use of the *chlorophyll* extracted from plants rich in this substance, such as parsley, spinach, etc.

The *aniline* greens are also employed.

To decide, then, as to whether the colour employed is of a poisonous nature or not, the substance should be thoroughly macerated in a small quantity of slightly warmed distilled water, and if the colouring matter can be dissolved out by these means it is in all probability organic and harmless, providing an insoluble colour is not a yellow created by gamboge. Most harmless colouring agents are soluble in alcohol, ether, or chloroform. The colouring matter being thus dissolved out, is next treated with a solution of sodium hypochlorite and gently warmed, when corroborative evidence of its organic nature is obtained if all the colour is discharged ("bleached"). Further, the ash of the substance will furnish no inorganic constituents that are capable of forming the colour employed. In this connection it should be noted that metallic mordants have been used for fixing organic colours.

The generality of coal-tar dyes will dye white wool without a mordant if the wool is heated in a solution of the dye.

If the colour is insoluble in water, and not bleached by

sodium hypochlorite, it is in all probability of a harmful nature, and of mineral origin, and the presence of copper, zinc, lead, chromium and arsenic should at least be tested for. Such tests, in many cases, are best made from a solution of the ash of the substance, upon the principles of qualitative and quantitative analyses which have been already dealt with.

Almost all the azo-dyes now so largely in use are decolorised by stannous chloride in an aqueous solution containing hydrochloric acid.

Of all articles, "tinned" vegetables have been most commonly found to be coloured (green) by copper. The coloration has been attributed to the formation of a copper salt by an acid derived from phyllocyanin, which body is very inert and insoluble in hydrochloric acid. Some contend that the copper—as it is converted into an albuminate—is non-injurious, because insoluble; but Dr. Charteris and others seem to have established the fact that the albuminate of copper is rendered soluble by the process of digestion.

Even expert opinion in this country still remains very conflicting as to the danger or innocuousness of the practice, as generally followed, *i.e.*, the peas are treated with a solution of cupric sulphate, this is almost immediately poured off, and the peas are subsequently well washed with water; they are next boiled in their tins and then soldered up. Probably the consensus of opinion tends to the direction of the innocuousness of the copper salt formed by the action of the copper on the peas (when this does not exceed  $1\frac{1}{2}$  grains per lb), both on account of its difficulty of solution, and from the very small quantities of copper in question.

W. Ogilvie and M'Lean Wilson separated the colouring matter yielded by copper to peas; and the former by experiments upon mice, and also upon his own body, showed that the organic salts of copper thus obtained were absorbed by the alimentary canal of man and mice, that they tend to accu-

multate in the liver, and are partially excreted in the urine, and probably to a less extent by the salivary glands. The latter experimenter found the copper compound to be soluble in distilled water, to which a drop of hydrochloric acid and a teaspoonful of Benger's liquor pepticus had been added, the whole being kept at a temperature of 98 deg. F. for several hours. It thus seems that the old theory that the copper formed with the legumen of the peas a compound which is quite insoluble by the gastro-intestinal secretions is erroneous.

## CHAPTER XIII.

## OTHER POISONS IN FOODS—ARSENIC IN WALL PAPERS, ETC.

ONE is sometimes called upon to examine an article of food for some poison, the nature of which may or may not be suspected ; in the former case a direct examination may be made for the suspected poison, but in the latter the more likely poisons must be sought after. The commoner poisons should be well understood by the Health Officer, and he should be able at any time to test for these, remembering in every case to retain one-third of the bulk of the substance under examination for any future contingencies that may arise.

It will frequently happen that the solution under examination is of a dark colour, or is of a somewhat slimy consistence, so that characteristic chemical reactions are unappreciable ; in these cases it is necessary to destroy the coloured and slimy matters before proceeding to test for metallic poisons, etc. This may be effected in the following way:—Some of the solution is treated with hydrochloric acid, and then placed upon the water-bath and heated for a short time. A solution of the chlorate of potash is then gradually added, until there is no longer any smell of chlorine, when the whole is diluted with about an equal bulk of distilled water and filtered. The filtrate can then be examined for the presence of poisonous substances.

There are several alternative methods of separating organic matter from inorganic:—

1. The organic matter may be destroyed.

(a) By careful ignition. But by this means the inorganic matter is apt to be also slightly affected. In addition to the losses indicated on page 78, *i.e.*, when the solid residue of a water is ignited, arsenic and antimony may escape, and copper, mercury, and zinc may suffer partial volatilisation. (The addition of concentrated sulphuric acid before ignition would, by forming copper and zinc sulphates, prevent the volatilisation of these metals).

(b) The substance is dried, finely divided, and placed inside a large crucible containing melting saltpetre in sufficient quantity that the melt remains pale in colour. There is no metallic loss by volatilisation in this method, and almost the whole of the inorganic matter is converted into compounds soluble in water.

(c) By heating with oxidising agents, such as a mixture of chlorate of potash and hydrochloric acid.

2. The organic matter may be extracted by a judicious selection of solvents (water, acids, alkalies).

3. The colouring or slimy matter may sometimes be separated by filtration, and dialysis is a useful expedient.

The mode of examining for arsenic and the commoner poisonous metals has been already seen, and it will only be necessary to introduce here a means by which phosphorus, strychnine, and "prussic acid" (hydrocyanic acid) may each be detected.

**Phosphorus.**—It is necessary that this metal be found in the free state, for when it exists in the form of phosphates its presence proves nothing—for phosphates are normal constituents of both animal and vegetable substances. The analysis must, moreover, be proceeded with without delay, or the phosphorus becomes oxidised to phosphoric acid.

The material suspected of containing phosphorus is put



into a flask, a cork is inserted (loosely) to the under surface of which a strip of filtering paper moistened with a neutral solution of argentic nitrate is attached, and the whole is heated to about 95 deg. F. In the presence of phosphorus the paper turns black, and if after some time no such change ensues, no unoxidised phosphorus is present.

If, however, there is a blackening, this may be caused by sulphuretted hydrogen, which may be tested for by a strip of paper moistened with a lead solution, and which may be absorbed by a concentrated solution of potash.

Valuable corroborative evidence of the presence of phosphorus is gained by testing the odour and luminosity of the substance.

If negative results are obtained it will be advisable to test for *phosphorous acid*, produced by the partial oxidation of the metal, and some of the substance should be transferred to an appropriate apparatus (Blondlot's) in which hydrogen gas is generated, and careful notice taken as to whether the emerald-green coloration of the otherwise colourless hydrogen flame, reveals the presence of phosphorus. If the flame remains uncoloured, organic substances may be present and preventing the colour from appearing; the apparatus should then be connected with an U-tube containing a solution of neutral nitrate of silver, through which the gas is allowed to pass for many hours in a slow stream; if phosphorous acid is present a precipitate containing the phosphide of silver will separate in the silver solution, and this precipitate should be tested for the characteristic flame.

**Hydrocyanic acid (HCN).**—This poisonous substance should be *early* sought after in any food stuff, etc., since it is extremely volatile and unstable. Another important poisonous cyanogen compound is the cyanide of potassium.

The ferro- and ferricyanides of potassium, and the cyanide of iron, are innocuous.

Prussic acid has an almond-like odour, but such is not in itself characteristic, for the odour of nitro-benzine and benzaldehyde closely resemble it.

If the article is not a liquid, it should be extracted with water, and the suspected solution filtered through a moistened filter-paper; the filtrate is collected, acidified with hydrochloric acid, and then tested with the perchloride of iron for *ferrocyanides*, and with ferrous sulphate for *ferricyanides* and *soluble thiocyanates*. The presence of the cyanide of iron is disclosed by the Prussian blue colour of the solution.

Should these all be absent then *poisonous cyanogen compounds* may be tested for. If guaiacum paper treated with a two per cent. solution of copper sulphate is suspended in the air over the suspected solution placed in a bottle, and the paper becomes blued when the whole is gently warmed, this is a strong indication of their presence; but the solution, if alkaline or neutral must be acidified by tartaric acid (Schönbein's test).

A corroborative test of great value is Scheele's iron test, in which an excess of potassic hydrate solution is added to the suspected solution; a mixture of a solution of a ferric and ferrous salt is then added, and a bluish-green precipitate results; after five minutes the whole is acidulated with dilute hydrochloric acid—the colour of Prussian blue results in the presence of hydrocyanic acid.

If the *innocuous cyanides* are *present* it is necessary to proceed thus:—If the solution is acid it should be rendered neutral by the addition of pure potassic hydrate solution, and subsequently to this, a cold concentrated solution of sodium bicarbonate is added. The mixture is then distilled, and

the distillate tested for hydrocyanic acid ; this distillation will exclude the presence of the harmless cyanides.

**Strychnine** is insoluble in ether, but soluble, with ease, in chloroform. Strychnine, brucine, veratrine and atropine may all be dissolved out by chloroform ; but strychnine may be isolated from these other alkaloids by taking advantage of its insolubility in cold absolute alcohol. Brucine and veratrine may be separated from atropine by making the solution alkaline and shaking with light petroleum (Dragendorff) ; the atropine is insoluble.

Strychnine may be tested for by dissolving the residue from the extract in a few drops of pure concentrated sulphuric acid, and if a little powdered chromate of potassium is further added as an oxidising agent, the solution acquires a blue-violet colour, changing after some time to wine red, and then to reddish-yellow.

In the case of **brucine** dissolve in concentrated nitric acid, and note the intense red colour that appears, and which subsequently becomes yellowish-red ; on adding stannous chloride to the solution, heated until it has become yellow, a most intense violet colour appears.

**Veratrine** dissolves to a pale yellow solution on treatment with concentrated sulphuric acid ; the colour gradually changes to a reddish-yellow, then to an intense blood-red, and finally to purple-red ; this lasts 2 or 3 hours, and then disappears. A little sugar sprinkled on to the yellow solution gives rise to a dark green colour, which gradually turns to an intense blue, and then slowly disappears. If continuously heated it sublimes unchanged.

**Atropine.** — Treat with a little fuming nitric acid, evaporate to dryness over the water-bath, and note any odour of hawthorn, and then add to the colourless residue, after it has cooled, a few drops of an alcoholic solution of potash and

resume evaporation—a violet coloration appears, which soon passes into red (Vitali).

**Arsenic.** — There are a few special points which have to be here considered, with regard to the presence of this very



FIG. 113.—Crystals of Arsenic.

poisonous metal in *food stuffs*—since a special line of procedure should be adopted in detecting its presence in these cases. The general practice is to stir the article up and mix it thoroughly with distilled water, and then place aside to settle; the almost clear supernatant liquid is then poured into a beaker; several of these washings are repeated, and ultimately all the water is drawn off as closely as possible. *Arsenious acid* is only sparingly soluble in water, but the washings should be carefully examined by Marsh's test; it is the residue which demands the closest investigation. Sometimes small hard white grains of arsenious acid may be picked out if a hand lens is used, and when these are placed in a glass tube and heated (after washing), a brilliant white sublimate forms, which when examined under the microscope is seen to consist of sparkling octahedrons and tetrahedrons; or again, small black scales of metallic arsenic may be seen, and when these are similarly treated, the well-known steel-grey arsenical mirror is formed upon the glass tube.

Arsenic sometimes gains admission to food even in the process of cooking, from the enamelled, etc., cooking utensils employed.

THE EXAMINATION OF WALL PAPERS, CURTAINS, DRESSES,  
ARTIFICIAL FLOWERS, CANDLES, ETC., FOR ARSENIC.

The dangers which may arise from the presence of this dreadfully poisonous agent in wall papers, etc., are too well appreciated, and have, unhappily, been too frequently demonstrated, to need any insistence upon here. There is a widespread impression among the general public that green is the only colour likely to contain this deadly agent; this has already been seen to be far from the truth, though there is no gainsaying that the metal has been found more commonly present in this colour than in any other.

The writer has detected considerable traces of arsenic in a paper of a mauve colour, in a house where, strange to say, the inmates had previously had a green wall paper removed on account of its supposed harmfulness. It is a difficult matter to enumerate the various colouring agents which may comprise arsenic in their constitution, and the matter may be dismissed by pointing out that almost every colour may do so; and it may be accepted that since the colour *green* is now so generally associated with arsenic in the popular mind, this is—of all others—the colour which is now most likely to be kept free from arsenic by manufacturers. Arsenical compounds are, however, sometimes used as mordants to fix the dye upon materials, in cases where the nature of the colour is quite harmless.

The metal is commonly dissociated from wall papers in the form of arseniuretted hydrogen by the action of the paste—especially when this is kept more or less damp by walls which transmit moisture. Small grains of arsenious acid also sometimes enter the atmosphere in a suspended form, and



rarely, even metallic arsenic may thus be discovered ; so that a microscopic examination of the dust of a room will sometimes disclose the presence of minute octahedral crystals and flakes.

The suspected article must be most thoroughly examined—much upon the principles shown upon a former page. If Scheele's green is suspected as furnishing the colour, a little of the paper or cloth may be thoroughly well soaked in ammonia, when a blue colour is created. In no case, however, can the employment of either Reinsch's or Marsh's test be dispensed with.

In applying Marsh's test, the paper or cloth, etc., is torn up into small pieces, and these are introduced into the apparatus.

Arsenic may exist in varying quantity in any material, *i.e.*, from a fraction of a grain (of arsenious acid) to even 80 grains per square yard—but since the smallest traces will condemn the article containing it, there is no necessity here to enter upon the subject of a quantitative analysis. It may be pointed out, however, that it is customary to estimate the metal as arsenious acid ("white arsenic" =  $\text{As}_2\text{O}_3$ ).

If negative results are got, advantage should be taken of the pattern upon a wall paper, curtain, or carpet, etc.; and when this consists of flowers and leaves, etc., of different colours, these should be cut out, arranged according to their tints, and scrapings or extracts of each colour should be separately introduced into the flask and examined.

The presence of arsenic in aniline dyes is sometimes exemplified by the appearance of a rash upon the skin of those whose underclothing is coloured by means of these agents. More especially is this the case with the scarlet and blue stockings so frequently worn, and the legs in consequence are the commonest seats of such eruptions.



The presence of **lead** in these various articles may also induce chronic lead poisoning, and large quantities are commonly contained in wall paints and papers, in floor cloths, and in artificial flowers—chiefly red and white. An examination, therefore, for the presence of this metal sometimes also becomes necessary.

## CHAPTER XIV.

## DISINFECTANTS.

THERE are numerous so-called "disinfectants" at present on the market, but few, under the usual conditions of employment, reach the standard of true disinfectants; many must be very little diluted if they are intended to kill any but the least resistant organisms, and none can have much effect as germicides when emptied down drains and sewers, where they either get too diluted, or very rapidly used up. Most of the disinfectants in use have marked *antiseptic* powers, as certified to by those who have examined them; but the term is misleading to the general public, who do not recognise the fact that an *antiseptic* may simply *inhibit the growth* of micro-organisms, whereas a *disinfectant* must be capable of *destroying* such organisms. The *antiseptic* power of many of these preparations is retained after considerable dilution, and they all either destroy or disguise odours more or less efficiently.

Disinfectants may be classified as:—

(a) Gaseous—such as sulphurous acid and chlorine.

(b) Liquid—such as those containing carbolic or cresylic acid, perchloride of mercury, chloride of zinc (Burnett's Fluid), ferrous sulphate, cupric sulphate, permanganate of potassium (Condy's Fluid).

(c) Solid—powders containing carbolic acid, chloride and hypochlorite of lime, bisulphite of lime, and lime.

Chlorine acts as an oxidiser by decomposing water and

liberating the oxygen, and Condly's Fluid, by readily parting with a portion of its oxygen, has similar properties. Sulphurous acid, bisulphite of lime, and ferrous sulphate, act as reducing agents by abstracting oxygen. Each of these agents is quickly used up in contact with organic matter, and much larger quantities are, therefore, required of them, than of substances like carbolic acid, which do not expend themselves.

Carbolic acid is obtained from the distillation of coal tar. It is very poisonous, and it is desirable that the sale of liquid preparations to the general public should be controlled and limited, and that the acid should be generally sold in carbolic powders. During the past 5 years about 400 deaths have resulted from the accidental or wilful drinking of liquid preparations of carbolic acid.

Each of the many disinfectants at present in use is claimed by the vendor to possess considerable disinfectant and antiseptic powers, and it is desirable that the Health Officer should be able to test these, and also to satisfy himself as to whether the proportions of active ingredients that the articles are stated to contain are actually present. There is no doubt that the carbolic preparations sold by other than the leading and most responsible firms, contain too little carbolic or cresylic acids to make them valuable for the purposes to which they are put, and many "carbolic powders" have been shown to possess little or no available carbolic acid; the poorer and more ignorant classes purchase large quantities of these worthless preparations. Many carbolic preparations contain a large percentage of water, homologous phenols and neutral tar oils. In some cases the base of carbolic powders is slaked lime (when the carbolate of lime formed is of little value as a disinfectant), in other cases the carbolic acid is added to silicious matter ("carbolicised silicate powders"), or to peat ("carbolicised peat powders"). It is very desirable

that in these powders soluble salts should be employed as absorbents of carbolic acid.

For the determination of the tar oils in crude carbolic acid the following simple method is given by A. H. Allen (*Commercial Organic Analysis*, Vol. ii., 545):—Introduce 10 c.c. of the sample into a graduated tube, and add gradually, noting the effect produced, twice its volume of a solution of caustic soda, free from alumina, and containing 9 per cent. of NaHO. Then close the tube and agitate well. The coal tar acids will be completely dissolved by the alkaline liquid, whilst, on standing, the neutral oils will form a separate stratum above or below the other, according as the admixture consisted of the light or heavy “oil of tar.” By the volume occupied by the oily stratum the extent of the adulteration is at once indicated. After noticing whether the tar oils are light or heavy, a volume of petroleum spirit, equal to that of the sample, may be advantageously added; its employment facilitates the separation of the oily stratum, and renders the reading of its volume more easy and accurate. Of course, the volume of the petroleum spirit used must be deducted from that of the total oily layer.

The percentage of phenols and cresols present in carbolic powders may be readily arrived at by the following means:—50 grammes of the powder are thoroughly shaken up in spirit, and thus all the free and available tar acids are abstracted; to this extract is added 50 c.c. of 10 per cent. caustic potash solution, heat is applied to drive off the spirit and to reduce the bulk of the liquid to about one half. If any tar oils separate out they are removed by filtration. The liquid having been emptied into a graduated cylinder, 50 per cent. sulphuric acid is added to neutralisation, when the tar acids separate out, and their volume can be read off; this volume,  $\times 2$ , will, of course, give the percentage amount. For the extraction of the total carbolic acid in powders containing lime,

50 grammes of the powder are cautiously and slowly treated with sufficient 50 per cent. sulphuric acid in a mortar, until a minute particle of the powder moistened with water gives an acid reaction when placed on litmus paper. Calcium sulphate is thus formed, and the carbolic acid is set free. The powder may then be exhausted with ether, and the ethereal extract distilled until the contents of the retort reach a temperature of about 230 deg. F. (when the residue will consist of crude carbolic acid); or the extract may be filtered into a flask containing 50 c.c. of 10 per cent. caustic potash solution; the contents of the flask are then well shaken up, and most of the ether driven off; the entire contents of the flask are next transferred to a separator—the lower layer that forms is drawn off, and the upper layer is washed with water, in the separator, and the washings added to the lower layer; then this is evaporated to about one half. The liquid is emptied into a graduated cylinder, and 50 per cent. sulphuric acid is added to neutralisation, when the coal tar acids that separate out  $\times 2$  will represent their percentage volume.

Good carbolic acid powders generally contain from 12—18 per cent. of crude carbolic acid, but they are liable to lose 1 or 2 per cent. by volatilisation. Half of the total oils in some powders consist of neutral tar oils (Allen).

For estimating the amount of ferrous sulphate, cupric sulphate, zinc chloride, etc., present in liquid preparations, it is convenient to calculate the amount of the salt present from a quantitative estimation of the metal.

The sulphurous acid contained in some disinfecting powders may be estimated by titration with decinormal thiosulphate after shaking up 1 gramme of the finely crushed powder in a large quantity of freshly distilled water. 50 c.c. of decinormal iodine is run in; the mixture is then made distinctly acid with dilute hydrochloric acid, and the excess of iodine is titrated with the thiosulphate. Each c.c. of the iodine solution reduced by the powder = 0.0032 gramme of  $\text{SO}_2$ .

Lowe's method is largely used for the purpose of making an approximate estimation of the amount of crystallisable phenols in liquid carbolic preparations, and for judging of the general quality of the preparation. 100 c.c. of the sample are placed in a retort (no condensing arrangement necessary), and the first portion of the distillate is slowly collected into a graduated tube—this will contain the water and 10 per cent. of the oils. If the oily matter floats on the water, light oil of tar is present; but it should be heavier than water, in which case it may be taken as containing about 50 per cent. of real carbolic acid. The next 62.5 c.c. of distillate is collected in a second graduated tube; it consists of anhydrous carbolic and cresylic acids in varying proportions. What remains behind in the retort consists wholly of cresylic acid, and still higher homologues of carbolic acid. The proportions of carbolic and cresylic acids in the 62.5 c.c. of distillate may be estimated by noting the temperature at which the mixture solidifies, and then, by a series of experiments, ascertaining what proportions of pure carbolic and cresylic acids must be mixed to give the same solidifying point.

The disinfectant and antiseptic powers of a "disinfectant" can only be gauged by direct experiments upon micro-organisms. In these experiments some of the most resisting organisms should be taken—such as the spores of *B. subtilis*, *B. mesentericus*, and *B. anthracis*, and *B. anthracis* itself; also some of the more common and important specific organisms should be experimented upon—such as the bacilli of typhoid fever, cholera, and diphtheria, and the streptococcus of scarlatina.

The organisms employed should be at the same time planted on some of the nutrient media used (nutrient gelatine, agar, etc.), in order to be certain that the organisms are active and typical, and the media satisfactory.

In testing the *disinfecting* powers of a preparation it is



diluted with water to varying amounts (1 in 5, 1 in 10, 1 in 100, 1 in 500, 1 in 1000, etc.), and a little of each strength is emptied into a series of test-tubes, which are then well shaken up with about  $\frac{1}{2}$  c.c. of stock culture of the different organisms selected. After an interval of 5, 15, 30 or 60 minutes, plate streak and stab inoculations are made by transferring a little of the contents of the test-tubes, by means of a sterilised platinum needle, to the nutrient media; the inoculated material is then placed in an incubator at 70 deg. F., and it is noted whether there is growth or not.

The *antiseptic* powers are tested on similar lines, but in this case various proportions of the disinfectant are mixed with the nutrient media, and these are subsequently inoculated; it is then noted whether the growth of the organism is inhibited or not.

It is a good plan to also experiment upon typhoid stools.

## PART VI.

### BACTERIOLOGICAL EXAMINATION OF AIR, WATER, SOIL, FOOD, CHOLERA EXCRETA, AND DIPHTHERITIC DEPOSITS.

By CHRISTOPHER CHILDS, M.A., M.D.Oxon., D.P.H.

To a text-book on "Public Health Laboratory Work" which deals almost entirely with chemical details, it appears desirable to append a brief treatise, setting forth the best and simplest methods at present available for the bacteriological examination of air, water, soil, and food. In the very short space afforded it is possible only to give such directions as may enable the student to make use of the elementary methods which he has acquired in the bacteriological laboratory.

It is assumed, therefore, that he has learnt how to prepare the various nutrient media required for the culture of micro-organisms, to sterilise apparatus and materials, to make "dilutions," pure cultures, and subcultures, to stain and mount samples from the cultures so produced—also that he has acquainted himself with the results obtained with stab, streak, shake, and plate-cultures of the more common organisms (pathogenic and non-pathogenic), and with the appearance and characteristics of such organisms. For more complete details of the methods of bacteriological analysis the student should consult the references which he will find given as foot-notes.

## EXAMINATION OF AIR.

For the purpose of studying the quality and quantity of micro-organisms contained in the air of any room or enclosed space, solid nutrient media, previously sterilised, may be exposed for a specified time in accordance with the following directions:—

1. Expose two Petri plates, each containing about 10 c.c. of nutrient gelatine (all previously sterilised), to the air of a laboratory or room; one for fifteen, the other for sixty minutes.

2. After the exposure cover each plate, place them in a damp chamber, and keep them for several days in the cool incubator (at 20 deg. C.), or in some place where the temperature will remain fairly constant between 15 and 20 deg. C.

3. Examine, and make sketches and notes of the appearance in each plate daily.

4. On the third or fourth day count the colonies on each plate with aid of a strong lens. Select representative samples of the various colonies, and from them make stab and streak cultures on gelatine and agar, and streak cultures on potatoes in Roux's tubes.

5. Observe, sketch, and make notes of the results produced; mount, stain and examine cover-glass preparations of the colonies under the microscope.

6. Identify, so far as possible, the micro-organisms thus isolated with the aid of such text-books as Crookshank's and Sternberg's *Manuals of Bacteriology*, Eisenberg's *Bakteriologische Diagnostik* (translated), Frankland's *Micro-Organisms in Water*, Woodhead's *Bacteria and their Products*, etc.

The above processes will be long, and somewhat tedious, but they should be repeated under varying conditions of time, surroundings, and locality. They will serve to give

some idea of the scarcity or prevalence of micro-organisms present in the air examined, to show the varying and widespread prevalence of such organisms in the atmosphere, and to prepare the student for the elaborate methods by which more accurate estimates may be made.

For the more exact determination of the number of micro-organisms, a known quantity of air (1 to 10 litres, or more) must be drawn through or over some medium in which all the micro-organisms present can be arrested and subsequently counted.

In Hesse's method\* an apparatus is used similar to that described on page 233 for the examination and estimation of solid particles suspended in the air. Instead of glycerine, however, a thin layer of nutrient gelatine is used for coating the interior of the tube. The tube, together with the gelatine, the caps and plug, are carefully sterilised, and are then ready for use. The air to be examined is drawn by an aspirator through the tube at the rate of about 20 litres per hour. In places where an excess of micro-organisms may be expected (*e.g.*, a crowded lodging-house) 1 litre or less may be sufficient for the examination. In the open air, 20 or more may be required. After the aspiration of a measured quantity of air, the apertures at the ends of the tube are closed with sterilised cotton wool and india-rubber cap respectively; the tube is placed in a cool incubator for three or four days. The individual colonies are then counted by means of Esmarch's apparatus.

From the number of colonies thus produced, the number of micro-organisms per litre or per cubic metre can readily be calculated.

The above process is somewhat cumbrous and costly. The

\* *Deutsche Med. Wochenschrift*, No. 2, 1884. Also *Phil. Trans.*, 1887, p. 62.

method employed by Cernelley and Wilson\* will be found more convenient. In their experiments Erlenmeyer flasks (of about 500 c.c. capacity) were used instead of Hesse's tube, the indrawn air being made to impinge on a thin film of Koch's peptone gelatine spread over the bottom of the flask, just as it impinges on the glycerine film in Pouchet's aeroscope (*vide* page 232), excepting that the inlet tube (which is of  $\frac{3}{8}$  inch internal diameter) is not drawn to a point, and extends only two-thirds of the way down the flask.

The outlet tube ( $\frac{1}{4}$  inch diameter) is bent round at the lower end so that it opens in the neck of the flask just under the stopper. It is open at both ends, and contains two cotton-wool plugs. The inlet tube is plugged with a glass stopper, fitted on with a piece of india-rubber tubing. The plug of the flask is firmly secured by copper wire, and the whole apparatus is sterilised by heating in steam at 100 deg. for an hour. The apparatus can be used at any time by unplugging the inlet tube, and connecting the outlet tube with an aspirator, by which the required quantity of air is drawn through the flask at the rate of about 20 litres per hour.

One advantage of the above two methods is that all the colonies formed from the micro-organisms which are arrested by the nutrient media can be examined *in situ*.

Various† other means have been devised for ensuring the complete arrest of the micro-organisms; *e.g.*, the required quantity of air may be aspirated through a measured quantity of sterile broth or water contained in a flask. A small fraction ( $\frac{1}{1000}$  to  $\frac{1}{100}$ ) of the fluid, after vigorous shaking, so as to diffuse the micro-organisms evenly, is then drawn out by a sterilised pipette, introduced into a tube of liquefied sterile nutrient gelatine, with which it is well mixed by

\* Proc. Roy. Soc., xliv., p. 455.

† Sternberg's *Manual of Bacteriology*; Miquel, *Les Organismes vivants de l'Atmosphère*, etc.

shaking, poured into a sterilised Petri plate, and placed in the cool incubator. On the third or fourth day the colonies are counted. The number of micro-organisms per cubic metre of air are calculated in the usual way. The micro-organisms may also be conveniently estimated with the aid of one of the soluble filters, first used by Pasteur, and since modified by Sedgwick and others;\* *e.g.*, by drawing the air through finely-powdered sugar in the following way:—A glass tube of about  $\frac{1}{4}$  inch in diameter and 8 inches long, drawn out to a point, and sealed at one end, is half filled with very finely-powdered dry white sugar. The sugar is kept in its place by a plug of cotton-wool, a second plug being placed in the open end of the tube. The tube, with its contents, is sterilised, allowed to cool, fixed vertically, and connected with an aspirator. The sealed (upper) end of the tube is nipped off with sterilised nippers, and the required quantity of air is drawn through the tube and sugar. The sugar is then shaken out into 50 c.c. of sterilised broth, from which small fractions (0.1 to 1 c.c.) may be introduced into nutrient gelatine plates, and examined and counted as in the preceding process.

The chief objection to these two methods lies in the difficulty of securing a perfectly uniform distribution of the organisms throughout the broth; for it is evident that if the organisms are crowded together, or the reverse, in the very small fraction of the broth which is abstracted for the estimation, very gross errors will result in the calculation of the total.

It must also be remembered that the colonies which are counted in the gelatine plates represent only those micro-organisms which have been able to develop in the time allowed, in the kind of medium in which they have been

\* Sedgwick and Tucker: *Boston Society of Arts*, 1888. Frankland, *Phil. Trans.*, 1887.



placed ; and under the conditions of temperature, aeration, etc., to which they have been subjected.

For instance, the spores of any anaërobic organisms which might happen to be present (*e.g.*, in a very dusty atmosphere), would not be developed at all under the methods described above. For the separation and estimation of the anaërobic organisms, the incubation must be carried on in vessels from which oxygen has been removed (*e.g.*, by potassium pyrogallate), or in which the air has been exhausted by an air-pump, or replaced by some other gas (*e.g.*, hydrogen) ; or at such a depth in a stab culture that the oxygen of the air cannot have access to the organisms, or by various other means.\*

To cultivate† and examine anaërobic organisms derived from the air, 10 to 20 litres of the air may be drawn through 50 c.c. of liquefied gelatine, containing 2 per cent. of grape sugar, in a Wolff bottle ; the bottle being half immersed in water which is maintained at a temperature of 38 deg. C. The inlet tube is then sealed by fusion. Aspiration is continued until the air is exhausted from the bottle, after which the india-rubber tube attaching the exit tube to the aspirator is firmly clamped. To ensure exclusion of air, all possible inlets are sealed with hard paraffin. The bottle is then placed in the cool incubator and examined daily for the appearance of colonies.

If any appear, roll tubes are prepared in the usual way. The roll-tubes, plugged with cotton-wool, are placed in a wide-necked flask, containing some moistened sand at the bottom, by which they are kept in an almost upright position. For the absorption of the oxygen a solution of pyrogallic acid is poured into the bottle. To this a 10 per cent. solution of

\* Sternberg's *Manual of Bacteriology*, p. 78.

† *Vide* Kanthack and Drysdale's *Elementary Practical Bacteriology*, p. 113.

caustic potash is added (10 c.c. for every gramme of pyrogallie acid).

The bottle is then exhausted, sealed, and placed in the incubator for several days. If colonies appear, deep stab-cultures may be made from them in gelatine or agar, containing 2 per cent. of grape sugar.

These subcultures can be placed in the wide-necked bottle, treated in the same way as that just described, and examined microscopically and by other methods.

This process has the advantage that several roll tubes can be incubated in the same bottle. Consequently it is especially useful in cases where the anaërobic organisms are scanty and diffused.

In other cases where the anaërobic organisms are more abundant, *e.g.*, in a very dusty atmosphere, or a fragment of soil, Fränkel's method,\* which is one of the simplest and most efficient, may be used.

An ordinary large test-tube may be employed, fitted with an india-rubber cork, through which two glass tubes pass, just as in an ordinary wash-bottle. The longer inlet tube passes through the sterilised and liquefied nutrient gelatine at the bottom of the tube, and serves to admit the hydrogen gas, which is passed through the apparatus in order to expel and exclude the air. The shorter tube gives exit to the gas.

Hydrogen gas, freed from oxygen by passage through an alkaline solution of potassium pyrogallate, is passed freely through the apparatus, bubbling through the liquefied gelatine. Care must be taken to ensure and ascertain by the usual method that all the air has been expelled, *viz.*, by collecting test-tubes full of the issuing gas until they no longer explode when applied to a light. The exit tube is then sealed by the blowpipe flame, and afterwards the inlet tube.

\* Fränkel, *Centralb. f. Bakteriöl.*, Bd. iii., p. 765.

The gelatine is liquefied, made into a roll plate in the same sealed tube, incubated at 22 deg. C. and examined daily.

The colonies which may develop can be counted, examined microscopically, and used for making subcultures.

### EXAMINATION OF WATER.

The bacteriological examination of water—quantitative and qualitative—is of considerable service for hygienic purposes. Thus Miquel's standards of purity,\* based on his large experience, may be regarded as an approximate indication of the degree of purity and pollution. These standards he summarises in the following table:—

		Bacteria per c.c.	
Excessively pure water	. . . .	0 to	10
Very pure	„ . . . .	10 to	100
Pure	„ . . . .	100 to	1,000
Moderately pure	„ . . . .	1,000 to	10,000
Impure	„ . . . .	10,000 to	100,000
Very impure	„ . . . .	100,000 and upwards.	

Of more reliable and practical use are the estimations of the relative number of microbes in large water supplies after filtration; since it has been shown that nearly all bacteria are removed by efficient filtration,† and that any serious defect in the filtration is speedily indicated by marked increase of the bacteria present.

The nature of the micro-organisms found in water also serves to indicate the source of pollution; and, as our methods and knowledge improve, we may hope for still further help in this direction. In several instances, again, the detection

\* Miquel, *Manuel Pratique d'Analyse Bactériologique des Eaux*, 1891.

† Frankland, *Micro-organisms in Water*.

of certain bacteria (*e.g.*, *B. typhosus* and *B. cholerae Asiaticæ*) may be of vital importance.

In examining the micro-organisms contained in water, especial care has to be given to its collection, in order to secure an average representative sample, and to prevent contamination with circumambient organisms. For estimation of the number of microbes contained in a certain volume—say 1 c.c.—of water, it is necessary to collect a sample—5 to 20 c.c.—in carefully sterilised glass vessels.

In the case of tap-water, where the sample may be examined on the spot, or carried to the laboratory without splashing, 100 c.c. may be collected in a sterilised flask, plugged with cotton wool, after allowing the tap to run for ten minutes.

To examine well-water it may be necessary to draw off the water for periods varying from a few hours to several days before an average sample can be obtained, since the number of micro-organisms may be enormously increased during stagnation.

In collecting samples from ponds, cisterns, rivers, lakes, etc., glass bottles with well-fitting glass stoppers may be used.

The bottles enclosed in metal canisters are sterilised at 150 deg. C. for 3 hours. To take a sample of water the bottle is immersed beneath the surface (in order to exclude any scum); the stopper is removed with sterilised forceps, and again replaced before bringing the bottle to the surface.

In some cases it may be more convenient to suck up the water in sterilised pipettes and transfer it at once to sterilised bottles.

For collection at various depths, or where the water is not easily accessible, Burgess's\* modification of Miquel's† apparatus may conveniently be used.

The neck of a 6 c.c. bulb is drawn out, and sealed in a

\* *Chemical News*, Aug. 3rd, 1894.

† *Manuel Pratique d'Analyse Bactériologique des Eaux*, 1891.

flame, after heating the bulb, so as to sterilise it and expel a considerable portion of the air which it contains.

To take a sample the bulb is fitted to a weighted support, suspended by a cord, which may be graduated in centimetres. The support is so constructed that by a sharp jerk of the cord a spring is liberated, which breaks off the neck of the bulb, thus admitting the required quantity of water at a depth which is measured by the graduated cord. The neck of the bulb is sealed by a blowpipe flame, and the sample can then be packed for transport.

All water samples should either be at once examined, or surrounded with ice, and examined as soon after collection as possible.

A sample of the water, free from adventitious organisms, having been collected, the number of micro-organisms per c.c. may be estimated in the following way:—

The sample is vigorously and continuously shaken—in order to diffuse the organisms as uniformly as possible.

Small quantities of the water (from 0.05 to 0.5 c.c.) are sucked up in sterilised pipettes, added to nutrient gelatine poured into Petri plates, incubated at 22 deg. C., and examined daily. On the third day the colonies are counted, and the micro-organisms per c.c. of the water calculated.

It must be remembered that the accuracy of the results depends in great measure on the uniformity with which the organisms are diffused throughout the sample under examination; also that, as in the case of air, the colonies produced in the gelatine plates represent only those organisms capable of developing under the conditions given of time, temperature, aeration, nature of media, etc.

For these reasons it is imperative to take care that the sample is well and thoroughly shaken, immediately before abstracting with the pipettes for inoculating the media; and, if at all comparable results are to be looked for, the nutrient



media should always have the same qualitative and quantitative composition and the same degree of alkalinity. Finally, the colonies should be counted at a certain time from the inoculation, preferably on the third day.

#### QUALITATIVE EXAMINATION OF WATER.

From the colonies, isolated in the gelatine plates, described above, subcultures may be prepared ; and notes and sketches should be made of the various organisms, as in the bacteriological study of Air. By repeating the experiments with water derived from various sources, *e.g.*, wells, streams, rivers, lakes, etc., the observer may make himself acquainted with the ordinary, and some of the extraordinary micro-organisms to be found in water.

The qualitative bacteriological examination of water is often of immediate practical importance, *e.g.*, when the spirillum of cholera is sought for ; or the *Bacterium coli commune*, and the *Bacillus typhosus*.

In order to test for the two last organisms in a sample of water, advantage is taken of the Chamberland or Berkefeld filter, by which the micro-organisms may be separated, and collected on the external surface of the filter.

A considerable bulk of the water—1 to 2 litres or more—is aspirated through a small Chamberland “candle,” previously sterilised in the autoclave.

A sufficient quantity of the filtrate (which is quite sterile) is poured into a sterilised plugged beaker.

The deposit of micro-organisms on the outside of the candle is brushed off, with a sterilised tooth-brush, into the beaker.

Of this mixture 1 c.c. is added to a series of tubes of carbolised gelatine and of carbolised alkaline beef broth (containing 1 per cent. peptone, and 0.5 per cent. sodium chloride).



The object of adding carbolic acid (viz., 0.05 per cent.) to the nutrient media is to inhibit the growth of the micro-organisms other than the *B. coli* and the *B. typhosus*, and more especially the liquefying bacteria.

Plates are prepared from the carbol-gelatine tubes, and incubated at 22 deg.; the broth tubes are incubated at 37 deg.

The plate cultures are examined daily for appearance of colonies characteristic of *B. coli* or *B. typhosus*, viz.:—  
“Flat crenated or irregularly outlined patches, thinner at the periphery than at the centre, translucent by transmitted light, greyish by reflected light; while colonies of both organisms growing beneath the surface, and imbedded in the gelatine, are rounder than surface colonies, and appear of a brownish colour by transmitted light.”\*

The difficulties of separating and distinguishing between these two organisms (*B. coli* and *B. typhosus*) are very great.

At present the following properties are mainly relied upon for distinguishing between them:—

1. The *B. typhosus* is the longer and more motile of the two.

2. The *B. coli* forms gas bubbles in gelatine shake cultures after 24 hours' incubation at 22 deg.; curdles milk in 1 to 2 days at 37 deg. C.; forms indol when grown in alkaline peptone broth† for 24 hours at 37 deg. C.

(The presence of Indol may be shown by adding to one of the broth tubes 1 c.c. of a 0.02 per cent. solution of potassium nitrite and a few drops of strong hydrogen sulphate, when a rose red coloration is produced). Cultures of *B. typhosus* do not produce these reactions.

The *B. coli* generally forms a streak of fæcal coloured

\* Klein: *Annual Report of Med. Officer of L.G.B.*, 1893-94, p. 77.

† *Vide Zeit. f. Hygiene*, Vol. vii., 1889, p. 581, and Vol. viii., p. 61.

slime on potatoes, the *B. typhosus* a colourless slime ; but these results are not absolutely constant. Most authorities, however, recognise the great difficulty of discriminating between these two micro-organisms, excepting in pure cultures ; especially as the *B. coli*, through its more rapid growth and stronger vitality, soon crowds out and obscures the more slowly growing *B. typhosus*.\*

DETECTION OF THE SPIRILLUM CHOLERÆ (Koch's "Comma Bacillus") IN SUSPECTED WATER.†

The difficulties of detecting the spirillum cholerae in suspected waters are often very great, since these spirilla are so frequently overgrown and obscured by other bacteria. Koch takes advantage of the fact (noted by Dunham in 1887) that the cholera bacilli multiply with exceptional rapidity in alkaline broth containing 1 per cent. of peptone and 0.5 per cent. of sodium chloride.

The growth collects on the surface of the fluid, as in the case of many other comma-shaped bacteria which occur in water.

To 100 c.c. of the suspected water, 1 per cent. of pure peptone and 1 per cent. of sodium chloride are added. The mixture should be made alkaline, if necessary, with carbonate of soda. This mixture is incubated at 37 deg. C.

At intervals of 10, 15, and 20 hours, microscopic examinations are made by preparing films from the surface of the fluid, whilst at the same time, plate cultures of agar, inoculated from the same source, are prepared and incubated at 37 deg. C. The resulting colonies are examined for comma-bacilli. If they are found they are used for inoculating fresh tubes, which

\* Frankland, *Micro-organisms in Water*, p. 267.

† *Der augenblickliche Stand der Cholera diagnose.* Koch, *Zeit. f. Hygiene*, 1893. (Translated by Duncan.)

may be tested for the "cholera-red reaction," and by animal inoculation.

Considerable doubt has been thrown on Koch's conclusions owing to the large number of other comma-shaped bacteria which have been discovered in various waters, and which, in several instances, share the properties supposed to be characteristic of true cholera. Koch, however, considers the presence of the true cholera bacillus in a water sample to be proved if, with the methods described above, comma-shaped bacilli are discovered which exhibit the cholera red reaction, and which prove rapidly fatal to guinea pigs when injected in very minute quantity into the peritoneum.

The "cholera-red reaction" is produced thus:—

To a peptone and sodium chloride culture (prepared as described above from one of the agar colonies), a few drops of pure hydrogen sulphate (absolutely free from any nitrous acid) are added. The appearance of a rosy red colour indicates the presence of the true cholera bacillus.

In order to obtain a reliable result with the above test it is necessary—

1. That the peptone used should be pure, and capable of giving the cholera-red reaction, since some of the commercial peptones fail in this respect.

2. That the culture examined should be a pure culture, since the cholera red may be produced on adding hydrogen sulphate to a culture which contains organisms of one kind, capable of forming indol from peptone, and organisms of another kind which reduce nitrates to nitrites.

#### DETECTION OF THE SPIRILLUM CHOLERÆ IN THE STOOLS OR INTESTINAL CONTENTS OF A PATIENT SUSPECTED OF CHOLERA.

In Koch's opinion a diagnosis of Asiatic cholera may be formed at once, in a large proportion of cases, by a skilled bacteriologist, on making a microscopic examination of

mucus flakes obtained from the stools or contents of the intestine. The bacilli stained by carbol-fuchsin are characterised by their comma-like shape, and especially by their peculiar grouping. "They form little heaps in which the single bacilli all have the same direction, so that it looks as if a little swarm of them were moving behind one another, like fish in slowly flowing water."

In all cases, however, the excreta must be further examined by making cultures, and submitting them to the tests described above for a similar examination of suspected water.

For this purpose one or more platinum loops of the excreta—or some of the mucus flakes, if possible—are to be added to a series of tubes containing the alkaline peptone broth, from which cultures must be prepared and tested, as in the case of the water examination.

#### EXAMINATION OF SOIL.

For studying the bacteria contained in soil, samples should be taken from various sources and at different depths. Surface soil may be scraped up with a sterilised knife.

From deeper layers soil must be taken from a fresh cutting in the earth, *e.g.*, one made for drainage, or by means of special borers, such as Fränkel's\* (*v.* page 175).

The samples should, without delay, be introduced into sterilised, plugged beakers. Minute quantities of a sample may be extracted in the loop of a stout platinum wire, by which they may be thoroughly mixed up with sterile liquefied gelatine in tubes and crushed; the particles being further diffused by careful shaking. From these tubes plates and roll tubes may be prepared, and the number of micro-organisms estimated as in the process of the bacteriological analysis of water.

\* *Zeit. f. Hygiene*, B. ii., 1887, p. 535.

Since most superficial soils contain enormous numbers of organisms, dilution of the gelatine is necessary before making the cultures; or the loops of soil may be shaken up with measured quantities of sterile water, from which, after subsidence of the sediment, cultures may be prepared and used for studying the organisms, and also plates for calculating the numbers present in a given quantity of soil.

A considerable proportion of the micro-organisms in soil are anaërobic—notably the bacilli of tetanus and malignant cedema.

Hence it is imperative to make anaërobic as well as aërobic cultures, for which purpose Fränkel's method (*v.* page 431) is convenient.

#### EXAMINATION OF FOOD.

In the bacteriological examination of food for hygienic purposes, the most usual object is to discover the nature of the micro-organisms, which may have given rise to a local outbreak of disease, the outbreak having been traced to some article of diet, which has been used in common by those affected; as, for instance, in "milk epidemics," and various outbreaks of gastro-intestinal disturbance.

For the examination of food suspected to be infected, the biological methods have been found to be by far the most effective and reliable.

In these methods animals are inoculated by feeding them with the suspected materials, or by injecting fluids or liquid cultures obtained from these materials.

The effects produced on the animals by these processes are carefully observed; and, after death, the organs and fluids of the corpses are examined microscopically, used for preparing cultures, etc., and again inoculated for the purpose of further examination.



Compared with these biological processes the ordinary bacteriological methods are at present very inadequate for the analysis of solid foods, owing to the accumulation in them of numerous micro-organisms, and the difficulty of isolating those which have given rise to dangerous symptoms in human beings.

It is desirable, however, that the student should make bacteriological examinations of the more common food stuffs and liquids by methods similar to those described for the analysis of water and soil especially.

#### EXAMINATION OF MILK.

The bacteriological examination of milk is more important than that of any other food, on account of its very general use, especially during infancy and childhood, the ages most susceptible to microbic disease; also of its constant liability to infection with micro-organisms, pathogenic and non-pathogenic, and of its special power of acting as a nutrient medium.

No micro-organisms have been detected in milk drawn from the udders of healthy cows under perfectly aseptic conditions.

But from the time when the milk leaves the udder it is subjected to contamination from numerous sources—from the udder itself, polluted by urine, fæces, and dirt, and, in diseased udders, by pathogenic organisms, *e.g.*, tubercle bacilli; from the hands of the milkman, the dust of the stall, the milk-pail, milk-can, and other vessels; from the air (during storage), and the water so frequently introduced for adulteration.

Consequently the micro-organisms are frequently to be counted in millions per cubic centimeter by the time when the milk comes into the hands of the purchaser.\*

\* Sedgwick & Batchelder: *Boston Med. and Surg. Journ.*, 1892.



Hence it is necessary to dilute the milk freely with sterilised water before making a quantitative bacteriological examination.

Similar dilution is also required in order to facilitate filtration when it is desired to separate off special organisms, *e.g.*, the *B. coli* and *B. typhosus*, by means of the Chamberland or Berkefeld filter.

The greater portion of bacteria present in milk, however, may be found in the sediment formed in a centrifugal separator, or after standing for 24 hours in a burette or separating funnel.

Consequently portions of this sediment may be examined microscopically for the bacteria mentioned above, as well as for micrococci, tubercle bacilli, and other pathogenic organisms; whilst other portions may be abstracted by the platinum loop for making cultures in the usual manner.

The presence of tubercle bacilli in milk may be especially tested for by examining the sediment produced in 50 c.c. of the sample, to which 10 c.c. of liquid carbolic acid have been added, after vigorously shaking the mixture, and allowing it to stand for 24 hours.\*

In examining this sediment microscopically, the films (after being fixed on the cover-glass) must be passed through a mixture of equal parts of alcohol and ether, and again passed three times through the flame before staining.

From the above statements it will be seen that, although the biological method is the most efficient for the bacteriological testing of milk, much may be learnt from simple microscopic examinations.

\* *Vide* Kanthack & Drysdale's *Elementary Practical Bacteriology*, p. 107.

## EXAMINATION OF THE THROAT FOR DIPHTHERIA BACILLUS.

The bacteriological diagnosis of diphtheria has become such a frequent and important process in hygienic work that it is thought desirable to append a brief description of the simplest methods available for this purpose.

To diagnose the presence of diphtheria bacillus in a suspected throat:—

1. Mop out the pharynx with a sterilised cotton wool swab, or, if possible, remove a fragment of the false membrane with a pair of sterilised forceps; introduce the swab or the fragment into a sterilised test-tube; plug the tube with sterilised wool; singe the plug and neck of the tube in flame.

2. Make cover-glass preparations from the membrane (after teasing), or by smearing the swab over the cover-glass; let dry and fix; stain with Löffler's methylene blue for a minute or two; examine with oil-immersion lens.

In this way the diphtheria bacilli may frequently be detected at once, being recognised by their typical appearance, viz., short rods (about two to three  $\mu$  long), more or less clubbed at both ends, frequently only at one; stained more deeply at the ends, often irregularly; disposed mostly in groups of 2, 3, or 4, which often resemble a collection of cuneiform letters, or a number of short, stumpy needles, which have been allowed to fall in small heaps on a table.\*

In some cases the diphtheria bacilli cannot be detected by these simple methods; and in all cases it is necessary that their presence should be confirmed by making cultures from the throat exudation, and examining these cultures for the characteristic bacilli.

For this purpose streak cultures should be made on sloped serum tubes, either with a fragment of the membrane, or

\* *Diagnostic Bactériologique de la Diphtérie.* L. Martin, 1894.

with the swab (which has been used for mopping the throat). The serum tubes are then incubated at 37 deg. for 24 hours.

If at the end of this time no colonies appear, and the method has been rightly carried out, there is no diphtheria bacillus in the material examined.

The diphtheritic colonies appear after about 14 or 15 hours' incubation, and may be recognised on the surface of the serum by their greyish-white colour, and regular, rounded outline; the colony appearing more opaque in the centre by transmitted light. It requires some skill and experience to distinguish these colonies for certain from those of other micro-organisms which frequently accompany the Diphtheria bacillus (notably strepto-cocci, staphylococci, etc.).

In all cases it is absolutely necessary to control the conclusions formed by making microscopic examination of the colonies in the same way as described above.

# INDEX.

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- Acarus domesticus*, 304; *A. farinæ*, 308  
Acidimetry, 167  
Actinomycosis, 356-357  
Adam's process for fat extraction, 272-275  
Aerated waters, 144-147  
Agrostemma githago, 315, 317  
Air bath, 12  
Air, composition of, 180-182; country air, 181; town air, 181-182; examination of, 194-236; carbon and compounds of carbon in, 182-183, 196-207, 213-220, 222, 243, 245; sulphur and compounds of sulphur in, 183, 220-221, 244-245; chlorine and compounds of chlorine in, 183, 221, 244-245; compounds of nitrogen in, 221, 244; organic matter, 183, 208-211; metallic fumes, 183; eudiometric examination, 184-190; eudiometric estimation of oxygen, 184-190; collection of samples, 191-194; ammonia, 212-213; marsh gas, 213; amount of gaseous impurities injurious to health, 222; ozone, 223-226; ozonometry, 226; aqueous vapour, 184, 226-228; peroxide of hydrogen, 229; suspended matter, 230-236; marsh air, 237; sewer air, 237-238; air over burial grounds, 238; air of mines, 239; air during fogs, 239; ground air, 239-241; scheme for detecting large quantities of gases, 242-245; bacteriological examination of, 426-432  
Aluminoid ammonia (*vide* Wanklyn's process)  
Alcoholic beverages, 366-374; proof spirit, 367; spirits, 367-369; beer, 369-371; finings, 369; wine, 371-374  
Alkalimetry, 165-167  
Allowable respiratory impurity, 206  
Aluminium for tinning foods, 402-403  
Alum in bread, 320-324; in baking powders, 316  
Ammonia in water (*vide* Wanklyn's process); in air, 212-213, 244; in coal gas, 253-254  
Ammonium sulphide in air, 220-221, 244-245  
Analytical form for coarse analysis of a mineral residue, 161-164  
Angus Smith's household test for purity of air, 207  
Aniline colours, 404-407  
Animal parasites, life histories of, 358-361

- Annatto, 284-285, 301, 405-406  
 Antimony in food, 404  
 Antiseptics, 419-424; employed in provisions, 399, 401  
 Aqueous vapour, 226-228  
 Arrowroot, 325, 332-333  
 Arsenic in water, 53, 58-60; in air, 183; in food, 399, 403, 415; in wall papers, etc., 416-417  
 Ascaris lumbricoides, 360  
 Aspergillus glaucus, 303-304  
 Aspirator, the, 209-210  
 Atmosphere (*vide* Air)  
 Atomic weights, 15-16  
 Atropine, 414-415
- Bacteriological examination of air, 426-432; water, 432-438; soil, 439-440; food, 440-441  
 Baking powders, 316-317  
 Barley-meal, 328, 332, 337  
 Barium in water, 53  
 Bean-meal, 327, 333-334  
 Beef, measly, 351-352  
 Beer, 369-371  
 Beggiotoa alba, 68, 129  
 Bell's process for estimating fat in milk, 270-272  
 Benzoic acid, 284, 401  
 Bilharzia hæmatobia, 360-361  
 Boracic acid, 283-284, 301, 401  
 Bothrioccephalus latus, 352, 359  
 Brandy, 367  
 Bread, 316-324; composition, 316; analysis, 317; adulteration, 318-319; microscopical examination, 319-320; alum in, 320-324  
 Brucine in foods, etc., 414  
 Bumping, during boiling, 97
- Bunsen burner, 1-2  
 Bunt, 310  
 Burette, graduated, 7-9  
 Burial grounds, air over, 238  
 Butter, 286-301; oleomargarine, 286-301; analysis, 287-299; microscopic examination of, 298-299; adulteration, 299-301
- Caffein, 382, 384, 386, 396  
 Calandra granaria, 307  
 Calcareous soil, 176-178  
 Calcium salts in water, 65-66  
 Carbolic acid, 419-421, 423; powders, 420-422  
 Carbonic acid, in water, 133-134, 145; in air, 182, 196, 243; in coal gas, 254  
 Carbon bisulphide, in air, 183, 221-222  
 Carbonic oxide, in air, 182, 213-220; in coal gas, 254  
 Carnelly-Mackie process, 211  
 Cereals, composition of, 311  
 Cheese, 302-305; adulteration of, 302-303; parasites in, 303-305; tyrotoxicon in, 305-306  
 Chemical balances, 2-5  
 Chicory, 382-385, 388  
 Chlorine, in water, 34; in air, 183, 245; as disinfectant, 419-420  
 Chocolate, 387  
 Chlorophyll, 407  
 Cholera bacillus, in water and stools, 435-437  
 Chromium, in water, 53, 61; in food, 404  
 Clark's process for softening water, 49-50

- Clark's scale of hardness, 47  
 Clay soil, 176, 178  
 Coal gas, in water, 31, 135; purification of, 246; composition of, 246-247; gaseous compounds of sulphur in, 248-253; ammonia in, 253-254; carbonic acid and monoxide in, 254; tar in, 254-255  
 Coal mines, air of, 239  
 Coccidia oviformes, 349  
 Cochineal, 405, 407  
 Cocoa, 385-387  
 Cocoa-nut oil, 300  
 Cœnurus cerebralis, 350  
 Coffee, 382-385, 387-389  
 — extracts, 385  
 Colorimetric analysis, 85  
 Colostrum, 263  
 Colouring agents in milk, 284-285; in butter, 301; in wine, 373-374; in sugar, 379-380; in sweets, etc., 403-408  
 Comma bacillus, in water, 437-438; in stools, 438-439  
 Condensed milk, 397  
 Condry's fluid, 419  
 Copper, in water, 52-57, 61-64; in tinned provisions, 399-403  
 Copper-zinc process for estimating nitrates and nitrites in water, 116-120  
 Corn, 307-312; parasites in, 307-311  
 Corn-coccle, the, 315, 317  
 Corn-flour, 326  
 Cotton-seed oil, 299-300  
 Country air, 181  
 Cows' milk, 256-285  
 Cream, 256-257, 266-267, 280-282  
 Cysticercus cellulosæ, 350-351; C. bovis, 351-352; C. pisiformis, 350; C. tenuicollis, 350  
 Dangerous water, sample of, 152  
 Darnel grass, 315  
 Decinormal solutions, 166  
 Deep well water from chalk, sample of, 158  
 Degrees of hardness (Clark), 47  
 Desiccator, the, 12-13  
 Diphtheria bacillus in deposits, 443-444  
 Disinfectants, 419-424  
 Distilled water, ammonia free, 89  
 Distoma hepaticum, 357-359  
 Dumas' process for estimating oxygen in air, 190  
 Dust brand in wheat, 309-310  
 Ear-coccle in wheat, 307-308  
 Earth-nut oil, 300  
 Effluents, sewage, 164  
 Eggs, 365  
 Ergot, 308-309  
 Eudiometer, the, 184  
 Eudiometric examination of air, 184-190  
 Expired air, 181  
 Facing tea-leaves, 394-396  
 Fat in milk, 270-277; in infants' foods, 397-398  
 Fehling's solution, 381  
 Finings, 369  
 Fish, 361-362  
 Flour, wheat, 312-315  
 Fog, air during, 239



- Food, bacteriological examination of, 440-441  
 Formalin, 401  
 Free ammonia (*vide* Wanklyn's process)  
 Frozen meat, 345  
 Fusel oil, 367-369
- Gamboge, 404  
 Garden vegetable mould, 177-178  
 Garrett's test for lead in water, 57  
 Gas burette, Hempel's, 185-187  
 Gaseous impurities in air, injurious amounts of, 222  
 Gases in water, 133-144  
 Gasparin's test for phosphoric acid in soil, 173  
 Gin, 367  
 Ginger beer, 145  
 Gluten, 313-314  
 Graham's condenser, 6  
 Graveyards, soil of, 179; air of,  
 Gravimetric analysis, 17, 64  
 Griess' test for nitrites in water, 114-115  
 Ground-air, 239-241  
 Ground-water, 169
- Hardness of water, 41-50; total hardness, 42-47; temporary and permanent, 43-44, 48; magnesium hardness, 49  
 Harvey's method of collecting gases from water, 142-143  
 Hehner and Richmond's formula for fat in milk, 272  
 Hempel's gas burette, 185-187
- Hesse's apparatus for collecting suspended matter of air, 232; for collecting ground air, 240-241; for bacteriological examination of air, 427  
 Honey, 381  
 Horseflesh, 362-363  
 Houzeau's test for ozone, 225  
 Hübl's iodine test, 296-297  
 Humanised milk, 398  
 Human milk, 259-260  
 Hydatids, 352-353, 359-360  
 Hydrochloric acid in air, 221-222, 244  
 Ignition of solid residue, 75-78  
 Indicators, 37; methyl-orange, 166-167  
 Infants' foods, 397-398  
 Iron in water, 29, 30, 52-57, 61-64; in food, 404  
 Kjeldahl's process, 174-175  
 Koettstorfer's test for butter fat, 291-293  
 Koch's comma bacillus, 437-439  
 Koumiss, 257
- Laboratory, fitting up of, 1-15  
 Lactometer, 267  
 Lactoscope, 276-277  
 Lard, 306  
 Lead in water, 51-57, 61-64; in air, 183; in food, 319, 399, 403; in wall papers, etc., 418  
 Lemonade, 145  
 Lentil flour, 327  
 Lie tea, 397  
 Liquoscope, the, 298

Lithia water, 144  
 — in water, 147  
 Litmus, 407  
 Living animal matter in water,  
 130-131  
 Living vegetable matter in water,  
 129-130  
 Logwood, 405, 407  
 Lolium temulentum, 315  
 Lunge and Zeckendorf, 203-204

Magnesium mixture, 71  
 Magnesium, hardness due to, 49;  
 salts in water, 66-67  
 Maize, 334, 337-338  
 Manganese, in water, 53, 61  
 Manures, 179  
 Mares' milk, 259-260  
 Margarine, 300  
 Marsh air, 237  
 — gas, 213  
 Marsh's test for arsenic, 58-60  
 Martius' yellow, 404, 406  
 Measly beef, 351-352  
 — pork, 350-351  
 Measuring flasks, 9  
 Meat, examination of, 339-365;  
 characters of good and bad  
 meat, 339-345; condemnation of  
 meat, 339, 345-348; tubercular  
 meat, 346-347; parasites in,  
 349-361; fish, 361-362; horse-  
 flesh, 362-363; sausages, pork  
 pies, etc., 363-364; eggs, 365  
 Melting point of butter fat, 297  
 Metallic fumes in air, 183  
 Metals, poisonous, in water, 51-64  
 Methyl orange indicator, 166

Metrical system of weights and  
 measures, 16-17  
 Mildew, 319-320  
 Milk, 256-285; composition of, 256;  
 of diseased cows, 260-261; analy-  
 sis, 262-278; adulteration, 279-  
 285; bacteriological examination  
 of, 441-442  
 Mineral waters, 144-147  
 Mines, air of, 239  
 Miniature gallon, the, 19-20  
 Miguel's standards of bacteriolo-  
 gical purity of water, 432  
 Moisture, in air, 184; in soil, 169  
 Molybdic solution, 70  
 Mucor mucedo, 304  
 Mustard, 376-377

Nessler's reagent, 87-88  
 Nesslerisation, 90-92  
 Nitrates in water, 110-123  
 Nitrifying organisms, 110, 175  
 Nitrites in water, 110-123  
 Nitrogen, oxidised in water, 110-123;  
 compounds of, in air, 221-222, 244  
 Normal solutions, 165-166

Oatmeal, 325, 334, 338  
 Offal, examination of, 343-345  
 Oidium abortifaciens, 308-309  
 Oidium aurantiacum, 317  
 Oleo-margarine, 286-301  
 Opinion on a water analysis, 25-26,  
 27-28, 147-150  
 Organic ammonia (*vide* Wanklyn's  
 process)  
 — carbon and nitrogen, 82,  
 106-109

- Organic matter in water, 80-84; in air, 183, 208-211
- Ova of parasites, 349
- Oxidisable organic matter in water, 99-105; in air, 211
- Oxidised nitrogen in water, 110-123
- Oxygen absorbed by organic matter in water, 99-105
- estimation of by eudiometry, 184-190
- by Dumas' process, 190
- Ozone, 223-226
- Ozonometry, 226
- Oxyuris vermicularis, 360
- Parasites, human, in water, 131; of cheese, 303-305; of corn, 307-311; of meat, 349-361
- Pea meal, 327, 333
- Peaty acids, in water, 33; in soil, 174
- Peaty soil, 177-178
- Peat, in water, 29, 155
- Penicillium glaucum, 319-320
- Pepper, 377-379
- Pepper brand, 310
- Peroxide of hydrogen in air, 229
- Pettenkoffer's estimation of carbonic acid in water, 133-134; in air, 197-202
- Phenol-sulphuric acid test for nitrates in water, 120-122
- Phosphates in water, 69-71; in infants' foods, 398
- Phosphorus, fumes in air, 183; in food, 411-412
- Piccini's test for nitrates, 113
- Picric acid test for nitrates, 120-122
- Plastering of wines, 372
- Poisonous products in food, 363-364, 399-400
- Poisons in food, 410-415
- Potassium in water, 146-147
- Poivrette, 377, 379
- Pork, measly, 350-351; pies, poisoning from, 363-364
- Potass water, 144
- Potato flour, 328, 332; in bread-making, 318
- Pouchet's aeroscope, 231-232, 428
- Preserved provisions, 399-409
- "Previous sewage contamination," 112
- Proof spirit, 367
- Provisions, tinned, 399-409
- Prussic acid in food, etc., 412-414
- Psorospermia, 356
- Puccinia graminis, 310
- Pure water, a sample of, 151
- Putrefaction, 343, 347-348
- Quantitative expressions used in water analysis, 18-20
- Rain water, 154
- Rainey's capsules, 356
- Ray-fungus, 356-357
- Reaction, of water, 32-33; of air, 195; of milk, 264
- Reagents required for water analysis, 14-15; milk analysis, 262
- Reichert's process for butter analysis, 293-296
- Report, water, 18-20
- Residue, solid, of water, 72-79
- Respired air, 181
- Rice, 329, 334, 338
- Richmond and Hehner's formula for fat in milk, 272
- River water, 156, 157

- Röse's test for fusel oil, 368  
 Round-worm, the, 360  
 Rum, 367  
 Rust, in wheat, 310  
 Rye meal, 328, 332, 337
- Sago, 326, 334  
 Salicylic acid, 284, 370, 372-373, 401  
 Saline ammonia (*vide* Wanklyn's method)  
 Sausages, 363-365  
 Schaffer and Wolpert's test for carbonic acid in air, 205  
 Scheme for quick analysis of water, 159-161  
 ——— detecting large quantities of gases in air, 242-245  
 Schlerostomum duodenale, 360  
 Schmidt's process for fat in milk, 275-276  
 Schultze's method of soil analysis, 171-173  
 Sea-water, 158-159; chlorine in, 36  
 Sedgwick's method of collecting bacteria from air, 429  
 Sediments in water, 33, 124-132; in milk, 264-265  
 Seltzer-water, 145  
 Semi-normal solutions, 166  
 Sesame oil, 300  
 Sewage, effluents, 96, 164-165; microscopical evidence of, in water, 127, 131-132  
 Sewage fungus, 68, 129  
 Sewer air, 237-238  
 Silica in water, 67  
 Skimmed milk, 285  
 Smut in wheat, 309 310
- Soap, test for hardness, 44-47  
 Soda-water, 144  
 Sodium in water, 146-147  
 Soil, chemical examination and analysis of, 168-179; bacteriological examination, 175, 439-440  
 Solid residue of water, 72-79  
 Sour milk, 265-266  
 Soxhlet's fat-extraction apparatus 274-275  
 Specific gravity, test for butter-fat, 289-291  
 Spectroscopic examination for carbonic oxide in air, 215-218  
 Spirits, 367-369  
 Sporondonema casei, 304  
 Spring water from chalk, 156  
 Starch, granules, characters of, 329-335; in infants' foods, 398  
 Strongylus filaria, 350  
 ——— paradoxicus, 350  
 Strychnine, test for, 414  
 Subsoil, 170  
 Sugar, 379-381  
 Sulphates in water, 68-69  
 Sulphuric anhydride in air, 183, 220-221  
 Sulphurous acid in air, 183, 220-221, 244; in coal-gas, 248-253; as an antiseptic in food, 401; as a disinfectant, 419-420, 422  
 Sulphuretted hydrogen, in water, 135, 140, 144; in air, 220-221, 245; in coal gas, 249  
 Suspended matter, in water, 78-79; in air, 230-236  
 Suspicious water, a sample of, 153  
 Sweets, colouring agents in, 403-408

- Tœnia, echinococcus, 352-353, 359-360; solium, 358; medio-canellata, 358-359  
 Tannin, in wine, 373; in tea, 397  
 Tapioca, 326, 334-335  
 Tar in coal gas, 254-255  
 Tea, 390-397; characters of the leaf, 393  
 Temperature, the, of water samples, 147; of air, 200-201  
 Temporary and permanent hardness, 43, 48-49  
 Thein, 390, 396  
 Theobromine, 386-387  
 Thermometer scales, 17  
 Thread-worm, the, 360  
 Tilletia, caries, 310; lævis, 310  
 Tidy's process in water analysis, 101-105  
 Thresh's test for dissolved oxygen in water, 136-140  
 Tinned provisions, 399-409  
 Tin, in water, 53, 60-61; in food, 399, 402, 404  
 Total solids, in water, 72-79; volatile and non-volatile, 75-78  
 Town air, composition of, 181-182  
 Trichina spiralis, 353-356, 361  
 Tricocephalus dispar, 360  
 Tubercular meat, 344, 346-347  
 Tuberculosis, bacillus of, in milk, 261, 265, 442  
 Turmeric, 284-285, 301, 405  
 Two-foot tube, 12  
 Typhosus bacillus, in water, 436-437  
 Tyrotoxon, 305-306  
 Uredo fœtida, 310.  
 — segetum, 309-310  
 Vapour, aqueous (*vide* aqueous vapour)  
 Veratrine, 414  
 Vibriones in wheat, 311  
 Vibrio tritici, 307-308  
 Vinegar, 375-376, 401  
 Vogel's test for carbonic oxide, 215-218  
 Volatile solids in water, 75-78  
 Volumetric analysis, 17  
 Wanklyn, Chapman, and Hall process, 85-98  
 Water analysis, opinion on, 25-28, 147-150; the physical characters, 28-33; chlorine, 34-40; hardness, 41-50; poisonous metals, 51-64; non-poisonous metals, 65-67; sulphates, 68-69; phosphates, 69-71; solid residue, 72-79; organic matter, 80-84; Wanklyn, Chapman, and Hall process, 85-98; oxidisable organic matter, 99-105; Frankland's process, 106-109; oxidised nitrogen, 110-123; water sediments, 124-132; gases in, 133-144; water samples, 151-159; schemes for water analysis, 159-161; bacteriological examination, 27, 432-438  
 Water bath, 10-12  
 — labels for samples, 26  
 — report, 18-20  
 — samples, collection of, 21-26  
 — standards, 148-149  
 Weevil, 307

Well-water, deep, from chalk, 158  
Weighing, operation of, 2-5  
Wet and dry bulb thermometers,  
226-227  
Wheat-flour, composition of, 312;  
analysis, 312-314; adulteration,  
314-315; microscopical char-  
acters, 331, 336  
Whip-worm, the, 360  
Whiskey, 367  
Winchester quart-bottle, 23

Wine, 371-374  
Wynter-Blyth's tube for sediments  
124-125  
Wood acid, 375  
Woman's Milk, 259-260  
  
Zinc, in water, 52, 53, 58, 62-63; in  
food, 399, 404  
Zymotic diseases of animals, and  
meat inspection, 348









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